

# PROCEEDINGS

SEMINAR ON

## SOIL AND LEAF ANALYTICAL METHODS THEIR TECHNIQUES AND INTERPRETATIONS

SERDANG, MALAYSIA  
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SEMINAR ON  
SOIL AND LEAF ANALYTICAL METHODS -  
THEIR TECHNIQUES AND INTERPRETATION

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SOIL MUSEUM

Edited by

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Organized by

The Malaysian Society of Soil Science

and

Soil Science Department, Universiti Pertanian Malaysia

## P R E F A C E

The Seminar on "Soil and Plant Analytical Methods - their Techniques and Interpretation" was jointly organized by the Malaysian Society of Soil Science and the Department of Soil Science, Universiti Pertanian Malaysia. Eighty-nine officers of various governmental and non-governmental organizations and the students of the Universiti Pertanian Malaysia attended the one-day Seminar held at the Universiti Pertanian Malaysia on the 9th December, 1978. A full list of participants appears in this report.

In general, the Seminar has achieved its goal in generating fruitful discussion on the current methods of soil and plant analyses and on the problems in the use and interpretation of the results obtained. It is happy to note that the Seminar also provided a venue for the Society's members from various fields of work to get acquainted and discuss their common problems. The President of the Malaysian Society of Soil Science in his welcoming speech expressed the hope that this meeting will be a permanent feature of joint activity between the Malaysian Society of Soil Science and the Department of Soil Science of the Universiti Pertanian Malaysia.

A.M. Mokhtaruddin  
H. Aminuddin

ORGANIZING COMMITTEE

Chairman: Sharifuddin Hj. Abd. Hamid

Secretary/Editor: Mokhtaruddin Ab. Manan  
Aminuddin Hussin

Members: Shamsuddin Jusop  
Mohd. Khanif Yusop  
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## OBJECTIVES AND THE PROGRAMME

### OBJECTIVES

The Malaysian Society of Soil Science and the Soil Science Department, Universiti Pertanian Malaysia have organized the Seminar with the following objectives:

- (i) to make agronomist, soil scientist and other practicing agriculturist familiar with and aware of:
  - a) analytical methods currently practiced in Malaysian laboratories
  - b) analytical facilities available in Malaysia
  - c) errors and precision on analytical calculus from Malaysian laboratories.
- (ii) to discuss the use and interpretation of the analytical results obtained for the various major crops in Malaysia.

### THE PROGRAMME

8.30 - 8.45 a.m. : Welcoming speech by Mr. Chew Poh Soon, the President of Malaysian Society of Soil Science.

Session 1 - Chairman: Dr. Othman bin Yaacob

8.50 - 9.10 a.m. : Soil chemical analysis in Malaysian laboratories  
- Sharifuddin Hj. Abdul Hamid

9.10 - 9.30 a.m. : Methods of leaf analysis in Malaysian laboratories  
- Poon Yew Chin

9.30 - 9.50 a.m. : Precision of soil and leaf analyses in Malaysian laboratories - M. Mohinder Singh and Norhayati Moris

9.50 - 10.10 a.m. : Tea break

10.10 - 10.30 a.m. : Routine determinations of soil physical properties in Malaysia

- Mokhtaruddin Ab. Manan

10.30 - 10.50 a.m. : Measurement of soil nutrient availability in Malaysia - Lau Chee Heng

Session 2 - Chairman: Dr. E. Pushparajah

11.15 - 11.35 a.m. : Problems in the interpretation of leaf and soil analytical data for hevea manuring

- K. Sivanadyan

11.35 - 11.55 a.m. : Interpretation of soil and leaf data for oil palm - Chan Kook Weng and Micheal Goh Khen Hing

11.55 - 12.15 a.m. : Problems associated with leaf and soil analyses techniques in relation to cocoa

- Thong Kar Cheong

12.15 - 12.35 p.m. : Potential use of soil and leaf analyses data for groundnuts and other field crops in Malaysia

- H.L. Foster and Ab. Rashid Ahmad

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WELCOMING SPEECH BY MR. CHEW POH SOON  
THE PRESIDENT OF MALAYSIAN SOCIETY OF SOIL SCIENCE

Ladies and Gentlemen,

This morning Seminar is organised jointly by the Malaysian Society of Soil Science and the Soil Science Department of the Universiti Pertanian Malaysia and on behalf of the organisers, I wish to welcome you all.

When discussing the programme of activities for the Soil Science Society at the beginning of this year, our Committee felt that some meetings where our members could take stock of current practices and views in various fields of soil science in this country would be useful. These meetings may also be good opportunities for members in other fields of work to acquaint themselves with their colleague's fields of interest.

After further deliberation, it was suggested that the Soil Science Department of the Universiti Pertanian Malaysia may be interested in organising the first of such meetings, and I am glad to say that the idea was enthusiastically taken up by Encik Sharifuddin, Head of Soil Science Department and his colleagues. The good work that you see this morning is largely their effort.

One of the important products of the meeting this morning is therefore the presence of the Universiti Pertanian Malaysia Soil Science students who are attending our meetings for the first time.

This morning, we will hear contributions from our members who are drawn from the Universiti Pertanian Malaysia, the Rubber Research Institute of Malaysia, Malaysian Agricultural Research and Development Institute and several private research organisations.

They have come forward willingly and at short notice to present their papers and I should like to thank them in advance for their very helpful co-operation.

This meeting has a special significance. We have representatives of almost every section of agriculture to present and discuss their work on analytical methods accuracy and interpretation here at the Universiti Pertanian Malaysia and we also have the future soil scientists and agriculturists here.

We hope that this meeting will be the first of many such regular meetings organised by the Malaysian Society of Soil Science and the Soil Science Department of the Universiti Pertanian Malaysia.

Thank you.

## **SESSION 1**

### **SOIL AND LEAF ANALYTICAL METHODS AND TECHNIQUES.**

## SOIL CHEMICAL ANALYSES IN MALAYSIAN LABORATORIES

by

H.A.H. Sharifuddin  
Universiti Pertanian Malaysia

## ABSTRACT

The methods and procedures of soil chemical analyses adopted by Malaysian Laboratories were surveyed. The survey showed that the soil chemical analyses carried out by various laboratories were basically similar, with some modification in certain laboratories.

Routine soil chemical analyses carried out by nine laboratories in Malaysia are discussed. These analyses include soil pH, organic carbon, total nitrogen, 'available' phosphorus, exchangeable potassium, calcium and magnesium, and cation exchange capacity. The analyses for total phosphorus, acid extractable cations, sulphates and chlorides, and soil conductivity are also discussed.

## INTRODUCTION

Soil chemical analyses in Malaysia are being conducted by three categories of laboratories, namely:

1. Government Departments: e.g. Department of Agriculture (DOA), Semenanjung Malaysia; DOA, Sarawak and DOA, Sabah.
2. Statutory Bodies: e.g. Malaysian Agricultural Research and Development Institute (MARDI), Universiti Pertanian Malaysia (UPM), Rubber Research Institute of Malaysia (RRIM), and Federal Land Development Authority (FELDA).

3. Private Agencies: e.g. Chemara Research Station (CRS), Dunlop Research Center (DRS), Highland Research Unit (HRU), Oil Palm Research Station (OPRS), and Agriculture Research and Advisory Bureau (ARAB).

These laboratories carry out the soil chemical analyses with the main objective of evaluating the nutrient status of the soil, so as to know the deficiencies or excesses that may exist, and this information form the basis on which fertilizer recommendations are made.

A survey on the methods used by all these laboratories shows that the soil chemical analyses carried out are basically similar, with some variation in certain laboratories. The summary of the methods used in these laboratories is as in Appendix 1.

The similarity in soil chemical analyses can be attributed to the effort made by the Sub-Committee on Standardization of Analytical Methods formed in 1966 by the Malaysian Society of Soil Science. This sub-committee has conducted cross-checks on soil analysis and has recommended standard procedures for the determination of soil pH, organic carbon, nitrogen, cation exchange capacity, exchangeable cations and easily soluble phosphorus to be adopted by the participating laboratories. Currently, the nine laboratories involved in these cross-checks are DOA Semenanjung Malaysia, DOA Sarawak, DOA Sabah, RRIM, FELDA, ARAB, CRS, HRU and OPRS.

The Standard and Industrial Research Institute of Malaysia (SIRIM) is also aware of the need to standardize the soil chemical analyses, and in 1974 established a Technical Committee on Methods of Soil and Plant Analyses. This committee is now in the process of producing the "Malaysian Standard Recommended Methods for Soil Chemical Analysis". Hopefully these recommended standard methods will be adopted by all laboratories in Malaysia.

The routine chemical analyses carried out by the Malaysian laboratories are soil pH, organic carbon, total nitrogen, 'available'

phosphorus, exchangeable K, Ca and Mg, and cation exchange capacity. Acid extractable cations, total phosphorus, soil conductivity, sulphates, chlorides and some other chemical analyses are performed by certain laboratories on request.

#### SOIL pH

All the laboratories in Malaysia adopted a 1:2.5 soil to solution ratio in measuring the pH of the soil, except ARAB laboratory which uses a soil to solution ratio of 1:5. Most laboratories measure soil pH in water as well as in 0.01 N KCl. The pH reading is taken after 16 hours.

#### SOIL ORGANIC CARBON

All the laboratories surveyed used the Walkley and Black procedure for the determination of organic carbon. In this method, 1.0 g of soil is used. The carbon in the soil is oxidised with 1 N potassium dichromate in concentrated sulphuric acid. The excess dichromate is back-titrated with a standard solution of ferrous ammonium sulphate, using diphenylamine as an indicator.

#### TOTAL NITROGEN

Total nitrogen is determined by the Semimicro-Kjeldahl method. The soil is digested with concentrated sulphuric acid in the presence of a catalyst mixture. The nitrogen in the sample is thus converted to ammonium ( $\text{NH}_4^+$ ) which is then determined from the amount of ammonia liberated by distillation of the digest with alkali. Dilute 0.01 N HCl is used to titrate the distillate.

In some laboratories autoanalyser is used to determine the ammonium-nitrogen colorimetrically, without having to distill the ammonia.

### ACID FLUOURIDE SOLUBLE PHOSPHORUS

The method that is employed in most Malaysian laboratories to determine the 'available' phosphorus is Bray and Kurtz No. 2. In this method, the combination of HCl and  $\text{NH}_4\text{F}$  will remove the easily acid-soluble forms of phosphorus, mainly calcium phosphate and part of the aluminium and iron phosphates. The  $\text{NH}_4\text{F}$  dissolves the aluminium and iron phosphates by its complex ion formation with these metal ions in acid solution.

2 g of soil is shaken for one minute with 20 ml of 0.03 N  $\text{NH}_4\text{F} + 0.1 \text{ N HCl}$ . The filtrate is then used to determine the phosphorus by the molybdenum-blue method.

From the survey, only UPM laboratory used different technique which also uses Bray and Kurtz No. 2 method but with 15 minutes shaking.

### EXCHANGEABLE CATIONS (K, Ca and Mg)

Exchangeable cations (K, Ca, and Mg) are determined in Malaysian laboratories by leaching the soil sample with 1.0 N ammonium acetate solution at pH 7.0. Generally, 10 g of soil is mixed with acid-washed sand and packed in a leaching tube between two layers of acid-washed sand. The soil is then leached for 5 to 6 hours with 100 ml 1.0 N ammonium acetate solution at pH 7.0. The concentrations of  $\text{K}^+$  sometimes  $\text{Na}^+$  in the leachate are determined by flame photometer and that of  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  by atomic absorption spectrophotometer.

All the laboratories used similar technique except ARAB, which employed shaking rather than leaching technique.

### CATION EXCHANGE CAPACITY (CEC)

The cation exchange capacity is determined by saturating the exchange complex with  $\text{NH}_4^+$ , and then determining the total absorbed  $\text{NH}_4^+$ .

The soil is first saturated with 1 N  $\text{NH}_4\text{OAc}$  at pH 7.0 (leaching for 5 to 6 hours, same as for the determination of exchangeable cations). The excess  $\text{NH}_4^+$  is leached with  $\text{KCl}$  or  $\text{K}_2\text{SO}_4$ . This  $\text{NH}_4^+$  is then distilled as ammonia and the quantity is determined by titrating with 0.01 N  $\text{HCl}$ .

All laboratories used this leaching technique except ARAB which shake and filter the soil suspension.

#### TOTAL PHOSPHORUS

Total phosphorus is determined by digesting the soil with 1:1 acid mixture of  $\text{HClO}_4$  and  $\text{H}_2\text{SO}_4$ . The phosphorus is measured colorimetrically by the molybdenum-blue method.

All the laboratories used similar method except that they differ in the weight of soil and the volume of acid used.

#### ACID EXTRACTABLE CATIONS

6 N  $\text{HCl}$  is being used by all laboratories surveyed to determine the acid extractable (total) cations. This analysis is used mainly to give an indication of the reserve K, Ca and Mg in the soil.

Generally, 2 g of soil is digested with 20 ml of 6 N  $\text{HCl}$ . The concentration of K, Ca and Mg are determined after Fe and Al are precipitated out.

#### SULPHATES AND CHLORIDES

The water soluble sulphates and chlorides are determined by shaking 100 g soil with 500 ml water. The sulphates in the soil water extract are determined by gravimetry as barium sulphate, and the chlorides by titration with silver nitrate in the presence of potassium dichromate solution.

## SOIL CONDUCTIVITY

The specific conductivity of the soil solution is usually measured when the presence of high concentration of salts in the soil solution is suspected. A soil to solution ratio of 1:5 is employed and the conductivity is measured after leaving the suspension overnight, using a suitable conductance bridge.

## OTHER CHEMICAL ANALYSES

On special request, some of the laboratories will carry out other chemical analyses such as lime requirement, exchangeable Al, free iron oxide, 'available' micronutrients, and total Si, Al and Fe in the soil.

## ACKNOWLEDGEMENT

The author is greatly indebted to the officers of the laboratories surveyed for supplying the information requested. A special thank goes to Mr. Lim Han Kuo of the Department of Agriculture Semenanjung Malaysia for his assistance.

The laboratories surveyed were:

1. Department of Agriculture Semenanjung Malaysia.
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5. Agricultural Research and Advisory Bureau (ARAB).
6. Chemara Research Station (CRS).
7. Dunlop Research Centre (DRC).
8. Highland Research Unit (HRU).
9. Oil Palm Research Station (OPRS).

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Singh, M.M., N. Moris and Z. Isa. 1978. Standardization of Soil Analysis. Paper presented at the Annual General Meeting of the Malaysian Society of Soil Science on 25th March, 1978. Kuala Lumpur.

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## APPENDIX I

## SOIL pH

All use 1:2.5 soil to solution ratio. pH is read after 16 hours. Exception: RRIM which read its pH (KCl) after 30 seconds.

## ORGANIC CARBON

All use Walkley and Black method

Weight of soil used: 1.0 g

Volume of 1.0 N  $K_2Cr_2O_7$  used: 10 ml

Volume of concentrated  $H_2SO_4$  used: 20 ml

Titration: 1/3 N Ferrous ammonium sulphate

Exception: OPRS which determines colorimetrically.

## TOTAL N

All use Semimicro-Kjeldahl method.

Weight of soil used: 0.25 g to 1.0 g

Volume of  $H_2SO_4$  used: 2 ml to 5 ml

Determination: By distillation and titration with 0.01 N HCl.

Exception: RRIM, OPRS and CRS which determine colorimetrically.

## "AVAILABLE" P

All use Bray and Kurtz No. 2

Weight of soil used: 2 g

Extracting solution: 20 ml 0.03 N  $NH_4F$  + 0.1 N HCl.

Shaking time: 1 minute

Exception: UPM uses 2.5 g soil to 50 ml extracting solution and shake for 15 minutes.

## EXCHANGEABLE K, Ca and Mg

All employ leaching technique (except ARAB)

Weight of soil used: 10 g

Extracting solution: 1 N  $\text{NH}_4\text{OAc}$  pH 7

Volume of extract used: 100 ml

Leaching time: 5 to 6 hours

Exception - OPRS uses 200 ml  $\text{NH}_4\text{OAc}$

- CRS leach for 4 hours

- ARAB uses 5 g soil and 20 ml  $\text{NH}_4\text{OAc}$ . The

suspension is shaken and leave overnight.

Filter and 15 ml + 15 ml  $\text{NH}_4\text{OAc}$  are added.

## CATION EXCHANGE CAPACITY

All employ leaching techniques (except ARAB)

Weight of soil used: 10 g

Extracting solution used:  $\text{NH}_4\text{OAc}$  pH 7

Leaching time: 5 to 6 hours

Washing of excess  $\text{NH}_4^+$ : 200 to 300 ml Ethyl alcohol (80 to 95%)

Displacement of absorbed  $\text{NH}_4^+$ : 0.1 N  $\text{K}_2\text{SO}_4$  or 0.1 N KCl

Titration: 0.01 N HCl

Exception: - OPRS uses autoanalyser instead of distillation and titration.

- ARAB follows a totally different method. To 2 g soil is added 8 ml  $\text{NH}_4\text{OAc}$ . Shake and leave overnight after filtering, 12 ml  $\text{NH}_4\text{OAc}$  is further added in batches of 2 ml and filtered. The rest of the procedure is the same as the others.

## TOTAL P

All use 1:1 mixture of concentrated  $\text{HClO}_4$  and  $\text{H}_2\text{SO}_4$

Weight of soil used: 2 g

Volume of acid used: 10 ml.

Final volume: 100 ml (HRU uses 50 ml)

Exception: CRS uses 1 g soil and 6 ml acid.

TOTAL K, Ca and Mg

All use 6 N HCl

Weight of soil used: 2 g

Volume of acid used: 10 ml

Final volume: 100 ml

Exception: OPRS uses 5 g soil and 50 ml acid. The  
final volume is 250 ml.

## METHODS OF LEAF ANALYSIS IN MALAYSIAN LABORATORIES

by

Poon Yew Chin  
Harrisons & Crosfield Oil Palm Research Station,  
Banting

## ABSTRACT

Large plantations and research organisations in Malaysia commonly carry out routine leaf analysis as a means of monitoring the nutritional status of the plant.

During the last two-and-a-half decades, the methods of analysis have improved markedly in both speed and accuracy with the introduction of atomic absorption spectroscopy and the autoanalyser. This has enabled laboratories to analyse large numbers of samples with a relatively small staff.

This paper discusses the analytical methods currently favoured by laboratories in Malaysia.

## INTRODUCTION

In Malaysia, at least seventeen laboratories carry out foliar analysis as a routine. Some of them are operated by Government and Statutory bodies whereas the majority of the laboratories are owned by commercial organisations, often attached to private research stations.

Leaf or foliar analysis is used widely as a means of detecting the early stages of, or trends towards nutritional deficiencies, toxicities and imbalances in plants. It also provides a guide for more discriminate fertilizer use for perennial and annual crops under continuous culture.

The procedure for leaf analysis can be said to be easier than soil analysis since total elements are normally determined whereas in soil analysis estimates can only be made of nutrients available for plant uptake in the short and long terms.

The choice of methods for leaf analysis depends primarily on the availability of equipment, staff, laboratory space and, most of all, the number of samples analysed annually. Laboratories which analyse only a small number of samples usually rely on manual methods, whereas those analysing large number depend more on automated instruments such as the autoanalyser and the atomic absorption spectrophotometer. The concept of atomic absorption spectra for elemental analysis was first suggested by Walsh in 1953 and today it is often the first choice method, especially for measurements at low concentration.

Whichever methods are adopted, the end result should be the same.

#### SAMPLE PREPARATION

Leaf samples are collected according to the procedure appropriate to the nature of the crop and the type of analysis required. For example, with mature rubber, low shade leaves are collected (Sharrocks, 1962) whereas with mature oil palms frond 17 is often sampled. The nutrient distribution along the palm front varies and it has been shown that a 20 cm sample of the mid-portion of the middle pinnae is most appropriate for defining foliar nutrient status (Chapman & Gray, 1949).

The leaf samples are sent to the laboratory as soon as possible after collection. This is to avoid growth of moulds and possible loss of dry matter (Sharrocks and Ratnasingam, 1962). Generally, the ideal sample size required by a laboratory is about 30 g oven-dried material. This will allow for replicate runs, analytical investigations, etc.

The mid-rib is removed if it is predominant like the oil palm. Some laboratories also remove the mid-rib in rubber leaves.

The leaves are cleaned on both sides with a clean flannel cloth soaked in deionised water and wrung out to just wet. Exceptionally dirty leaves (especially those collected during long dry periods) are first cleaned with a solution of 0.2% phosphate-free detergent followed by deionised water.

The leaves are then cut into small pieces and dried in an air-draught oven at  $70\pm10^{\circ}\text{C}$ . The period of drying depends on the nature and size of the material, but is usually complete after 16 hours (overnight). The sample is considered dry when it is crisp to the touch, and quite brittle. Recently, attempts were made to dry oil palm leaves in a microwave oven. Preliminary results have indicated no differences in major nutrient contents in the leaf as compared to the conventional oven drying method.

The dried sample is passed through a grinding mill fitted with a 1 mm sieve and the ground sample is stored in a screw capped bottle. The ground sample is redried at  $105^{\circ}\text{C}$  for 2-3 hours, capped while hot, and stored in a dessicator.

#### DRY ASHING

Ashing is normally performed in silica dishes in a muffle furnace. Two laboratories carry out ashing in aluminium dishes. The temperature is maintained at  $300^{\circ}\text{C}$  for about 1 hour or until smoking ceases and then raised to  $500^{\circ}\text{C}$ . The temperature is maintained at  $500^{\circ}\text{C}$  until a white or greyish-white ash is obtained. This may take a few hours. When the sample size is large and several of the samples are shed together in a furnace, overnight ashing may be more convenient.

The ash is treated with concentrated hydrochloric acid and slowly evaporated to dryness on a water bath. This hydrolyses

the pyrophosphates and dehydrates the silica. 20% nitric acid is added and digested on a water bath for 1 hour. The solution is filtered and made up to volume (Ash solution).

#### CURRENT LABORATORY METHODS

For convenience a flow chart of leaf preparation and analysis as adopted at the Harrisons & Crosfield Oil Palm Research Station is shown in Appendix 1.

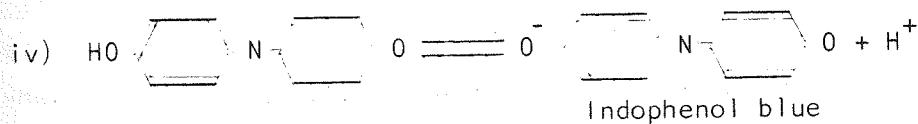
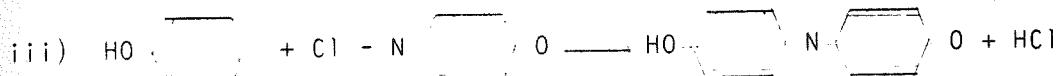
The analytical methods used are outlined below:

##### Nitrogen

Protein-N in leaf is commonly measured. This is achieved by heating 60 mg of the leaf material with 2 ml concentrated sulphuric acid in the presence of 1 g of a catalytic mixture of sodium or potassium sulphate and selenium, until an almost colourless solution is obtained. The digestions are carried out in test tubes or in conventional Kjeldahl flasks. The amount of acid and catalytic mixture added is important as this affects the digestion temperature. Bremner 1965, claimed that nitrogen is lost when the digestion temperature exceeds 400°C, and this temperature is attained when the concentration of potassium sulphate is about 1.3 to 1.4 g per ml of sulphuric acid. All laboratories in Malaysia use a ratio of less than 1 g per ml.

The method of determination of the ammonium is either by distillation or by colorimetry. In the distillation method, the ammonia is distilled into boric acid and then titrated with standard sulphuric acid. With the colorimetric method, ammonium ions react with alkaline phenol and hypochlorite, and on warming, an intense blue colour is produced, which is closely related to that of indo-phenol dyes. The latter is measured with the aid of the autoanalyser (Varley, 1966).

Apparently no rigorous proof on the mechanism involved in the formation of indophenol blue is known but Tetlow & Wilson 1964, citing Bolletar *et. al.*, have suggested the following reaction scheme:



In Malaysia, nine laboratories adopt the colorimetric method of determination and the other eight the distillation method.

#### Chloride

The leaf sample (1 g) is mixed with a basic medium such as calcium oxide (0.25 g), made into a paste with a few drops of water and ashed in a muffle furnace at 500-550°C. A basic medium is necessary to prevent volatilization of the chloride. The ashed sample is digested with hot water, neutralised with dilute acetic acid and the chloride determined by titration with silver nitrate.

#### Sulphur

The leaf material (2 g) is digested with concentrated nitric and perchloric acid (Middleton, 1962). The digest is neutralised with ammonium hydroxide, acidified with hydrochloric acid and determined gravimetrically as barium sulphate. Some laboratories prefer the digestion method of Cunningham 1962 in which the plant material is digested with fuming nitric acid and ashed in the presence of magnesium nitrate. The precipitated barium sulphate is dissolved in ammonium EDTA and barium measured by atomic absorption.

### Molybdenum

The leaf material is wet ashed with nitric/perchloric acids and molybdenum measured colorimetrically either by the thiocyanate coloured complex or the molybdenum-dithiol complex methods (Sandell, 1965).

### Phosphorus

All the laboratories in Malaysia measure phosphorus in leaf from the 'ash solution'. Phosphorus is measured as phosphate by the vanadomolybdate method (Kitson & Mellon, 1944). The advantage of the method is that the colour of the yellow vanadomolybdate-phosphate complex is stable and is relatively free from interferences (Chapman & Pratt, 1961).

The instrument used for the determination is either the autoanalyser or a simple colorimeter and the wavelength selected is normally between 400-420 nm.

### Potassium and Sodium

All the Malaysian laboratories use flame emission for determination of potassium and sodium in leaf material. The 'ash solution' of the plant material is passed directly into the flame and the concentration obtained by comparison with standards. Laboratories which use the flame photometer unit of the autoanalyser introduce lithium (as lithium nitrate) to act as an internal standard.

### Calcium

Atomic absorption spectroscopy is the method most commonly used for the determination of calcium in leaf material. The various atomic absorption instruments available are often very sensitive and hence the ash solutions have to be diluted several fold before the determination.

With an air-acetylene flame, calcium determination is prone to chemical interference attributable to the aluminium, silicate, phosphate and sulphate ions commonly present in leaf material. Interference is prevented by the addition of a radiation buffer (releasing agent) such as strontium or lanthanum (David, 1960).

Only three laboratories in Malaysia use the EDTA titration method whereas the rest use the atomic absorption.

#### Magnesium

Two laboratories use the EDTA titration method, one the thiazole yellow colorimetric method (Young and Gill, 1951) while the other laboratories in Malaysia measure magnesium by atomic absorption. The presence of a radiation buffer is necessary as the interfering ions that affect calcium analysis by atomic absorption similarly affect magnesium.

Three laboratories which have an atomic absorption also adopt, as an alternative, the Eriochrome black T (E.B.T.) colorimetric method on the autoanalyser.

#### Manganese

Atomic absorption is commonly used in the determination of manganese in the 'ash solution'. However, four laboratories use either the periodate or the formaldoxime colorimetric method (Sandell, 1965).

Apparently high iron interferes in manganese determination by the formaldoxime method, leading to enhanced values. The high iron in leaf is uncommon and has only been observed in sugar beet, the iron content of which can be as high as 4,000 ppm. Under such circumstances determination by atomic absorption is necessary. In Malaysian crops such as rubber and oil palm where the iron is normally about 100 ppm such problems do not arise.

### Boron

Ten laboratories favour the carmine colorimetric method (Hatcher & Wilcox, 1950), though two laboratories prefer the curcumine and quinalizarin methods. One laboratory uses the azomethine H colorimetric method on the autoanalyser.

In the carmine method, the ash is digested with 6 N hydrochloric and the 'ash solution' reacted with carmine reagent in concentrated sulphuric acid. The blue carmine/boron complex is then measured with a colorimeter at 610 nm. Since the carmine/boron complex is developed in a highly corrosive medium, the autoanalyser is not used by any Malaysian Laboratory because of the possible dangers involved. Other laboratories have been known to use the method on the autoanalyser by having acid flex tubes.

As a precaution against contamination of boron by pyrex glassware, all containers used are either of heavy duty polythene or soft (soda) glass.

### Iron, Copper and Zinc

Leaf material is commonly dry-ashed and the metals determined by atomic absorption.

Three laboratories determine iron by the dipyridyl or tiron colorimetric methods. One laboratory measures copper and zinc colorimetrically by diethyldithio carbamate and zincon respectively (Chapman and Pratt, 1961).

### COMPARATIVE LABORATORY ANALYSES

As a means of monitoring the accuracy of analysis of the various laboratories in Malaysia, cross-checks on common Malaysian plant materials are held regularly. The general order of agreement obtained during the period 1975-77 by sixteen Malaysian laboratories is shown in Table 1.

Table 1

Coefficient of Variation (%) for Plant \*  
Analysis by Sixteen Malaysian Laboratories

Elements	C.V. %
N, P	0- 5
K, Ca, Mg, Mn	5-10
Fe	10-15
Cu, Zn, B	15

\* Results quoted from Dept. of Agric.  
publication of June 1978 on "Local  
cross-checks on Plant Analysis 1975-77".

The high coefficients of variation especially for iron, copper, zinc and boron may be due to the fact that most of the laboratories do not carry out the analysis as a normal routine but only occasionally. Low values of trace metals in the leaf may also contribute to the high recorded coefficients of variation.

Though the results appear satisfactory, there is certainly room for improvement. This can be brought about by examining in detail the various test procedures adopted by the different laboratories in Malaysia. This could prove quite onerous because it would be necessary to consider even the most minute details which are inherently different between laboratories. It may be appropriate to mention that within laboratory error is often very small.

Chemists do monitor the accuracy of their laboratories regularly with cross-checks and it is hoped that results which persistently differ from the general mean will be looked into by those concerned.

## ACKNOWLEDGEMENT

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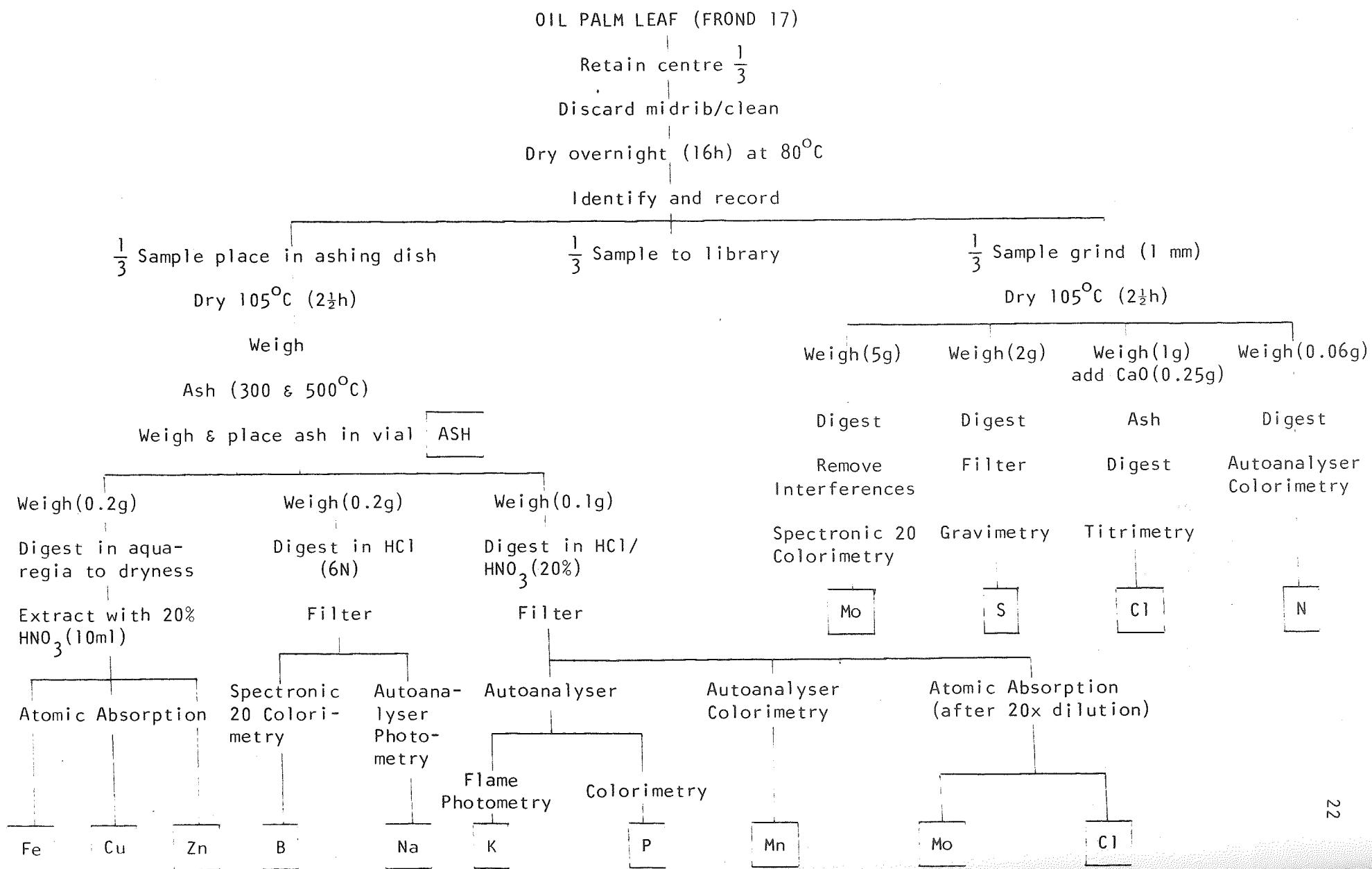
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APPENDIX I

METHOD OF OIL PALM FOLIAR ANALYSIS



## PRECISION OF SOIL AND LEAF ANALYSES IN MALAYSIAN LABORATORIES

by

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### ABSTRACT

Studies on the precision of soil and leaf analyses in Malaysia are reviewed. Field sampling errors are shown to be far more significant than laboratory errors. Laboratory errors are mainly attributed to chemical treatment or digestion procedures. Standardization of some of the analytical procedures has brought about some improvement in soil and plant analyses but there is still appreciable interlaboratory variability particularly in soluble P, total P, exchangeable Ca and Na and coarse sand in the case of soil analysis and the minor elements, particularly copper, manganese and zinc, in leaf analysis.

### INTRODUCTION

Soil and leaf analyses are now widely used in Malaysia for assessing fertilizer requirements of crops on different soils. In 1977, the laboratories of the Rubber Research Institute of Malaysia analysed some 4,000 soil and 28,000 plant (mostly foliar) samples requiring 77,000 and 124,000 determinations respectively. The number of laboratories which carry out soil and plant analyses in Malaysia have increased rapidly in the last decade. This clearly indicates the growing acceptance to make fertilizer assessment more quantitative and reliable. It is therefore important that the soil and leaf analytical methods used should be reliable. The exercise on standardization of soil and plant analyses in Malaysia carried out by local

laboratories since 1966 clearly indicates that this need to make laboratory analysis more precise has been duly recognised.

## SOIL ANALYSIS

Soils being heterogenous and complex in nature pose difficulties in defining their chemical properties precisely. The precision is dependent on several factors which may be broadly classified into soil sampling errors and analytical errors.

### Field Variability and Soil Sampling Errors

It is a fact that tropical soils being highly weathered, can be very heterogenous even over small distances, particularly in respect of those soil properties which influence markedly agricultural management practises. Ng and Ratnasingam (1970) showed that there is considerable variation in nutrient contents within a 10 ha field on the same soil series and at least 30 random cores were required to achieve results for phosphorus, potassium, magnesium and calcium within 20% of mean values on Batu Anam, Prang and Selangor series soils. To achieve a better precision of 10%, as many as 120 sampling points were found required in these soils. In a further study involving 6 other soil series (Rengam, Munchong, Malacca, Kuantan, Klau, Serdang), Ng *et. al* (1971a) examined 3 methods of sampling, namely normal sampling consisting of 1 composite sample from 10 different points, 40 separate samples from 1 point each per 0.4 ha and 40 composite samples from 3 points each per 0.4 ha. In terms of precision, the 3-point method gave lower variability. pH was least variable (c.v. < 10%) while C and N had c.v.'s in the range 10-20%. The other parameters (total P, acid fluoride P, acid-extractable cations and exchangeable cations) gave much higher variabilities, sometimes even above 100% c.v. (Table 1). They concluded that for pH, 2 sampling points were sufficient for 10% precision but for C and N, 20 sampling points were required. For the other parameters, at least 30 sampling points were necessary for a precision

of 20% but even at this intensity of sampling, 20% precision is not necessarily obtained for all the parameters for the different soils (Table 2).

#### Analytical Errors

The errors of laboratory analysis may be broadly classified as determinate errors (sub-sampling, processes such as heating, extraction, filtration, instrumental measurement) and indeterminate errors (operator, working conditions). Determinate errors can be estimated and reduced in magnitude but indeterminate errors are more subjective and less orderly.

Ng *et. al* (1971b) examined the total error variation of routine chemical analyses for its components attributable to operator, day, chemical treatment or digestion and instrumental measurement on ten common soil series. They observed that in the case of pH, C, N, acid fluoride P, exch. K and Mg and total P and Mn, the total error variation was below 8% c.v. In the case of acid extractable (6 N HCl) cations and exch. Ca the c.v. was 10-20%. Instrumental and day variations were found to be relatively small and operator differences contributed occasionally. Chemical treatment was the major source of the total laboratory test error (Table 3).

The rather large errors found in the determination of acid extractable cations are associated with variable conditions of extraction such as temperature, duration of heating and variable loss by absorption of the cations by the gelatinous precipitate of iron and aluminium hydroxides.

#### Inter-Laboratory Cross-Checks

In order to improve precision in soil analysis, local laboratories have undertaken a series of cross-checks since 1966. Some standardization of test procedures has been progressively achieved. Table 4 summarises the cross-checks carried out so far and the number

of laboratories which have participated. The number of participating laboratories have increased from four to nine.

Table 5 summarises the variability observed between the laboratories from 1969 to 1975 (Singh, et.al, 1978). The results show that the analyses can be classified into 3 groups:

Group A	pH	good agreement
Group B	C, N, CEC, Exch. K, Exch. Mg, Conductivity, Clay, Silt, Fine Sand	c.v. up to 30%
Group C	Soluble P, Total P, Exch. Ca, Exch. Na, Coarse Sand	High variability

The agreement between the laboratories for pH ( $H_2O$ ) and pH (KCl) has been good throughout the cross-check exercise. This is partly due to the simple and precise nature of the analysis. In Group B, the variability of the analysis was as much as 30% c.v. The parameters in this group include C, N, CEC, exch. K, Mg, conductivity, clay, silt and fine sand. The agreement for C, N and total P can be considered to be relatively satisfactory especially in the later years where the variability has decreased. This can be attributed to several factors, namely minimisation of pretreatment errors (arising from subsampling and grinding) by despatching ground samples from one source for C and N analysis, standardisation of digestion procedures and methods of determination for N and P. Previously some laboratories used automated colorimetric procedures for N while others used the kjeldahl distillation method. This situation was also true for total P analysis. With standardisation in the type and volume of digestion mixture used and adoption of the molybdate-blue method for quantitative estimation of P, the determinative sources of errors have been minimised, resulting in improved precision and agreement within the laboratories. However, steps to improve the variability among the laboratories should be continued as the variability is still rather high.

For conductivity measurements, several sources of errors contribute to the rather high variability observed. In their report, Ahmed et. al (1971) showed the time effect on conductiveness of 1:2.5 and 1:5 soil:water mixtures, and on the use of stirring and shaking methods to facilitate the reaching of equilibrium. Also correction for temperature variation has to be taken into account.

Mechanical analysis has been observed to give rather high variability. To improve the poor agreement within the laboratories methods of mechanical analysis were standardised in the 1971 cross-check (Singh 1971a). A standard sieve size of 0.21 mm was used to separate the coarse from the fine sand and sampling for clay at 6 cm depth after 4½ hours and drying of silt and clay fractions overnight at 105°C. In the 1975 cross-check, only clay and silt tended to show slightly better agreement within the laboratories with variability of about 10% c.v. for silt and around 17% for clay. Very high variability was observed for coarse sand.

Rather poor inter-laboratory agreement in the determination of CEC and exchangeable cations namely Mg, K, Na and Ca was observed since the initial stages. Variability of CEC ranged from 15.6 to 32.8% c.v. while that of the exchangeable cations from as low 11.3% to as high as 43.6%. The leaching step contributes to the main source of error in the determination of CEC and exchangeable cations plus an additional error incurred during the washing step for CEC determination. Efforts to improve the extraction and subsampling steps were undertaken by the sub-committee. Several ratios of soil to washing solution to leaching solution have been suggested and tried out in the 1975 and 1977 cross-check exercises, i.e. 1:10:10; 1:20:10 and 1:20:20 ratios. Results of this exercise, however, have not been discussed yet. For the exchangeable cations it was observed that variability was generally greater when the levels were low (below 0.1 me %).

Soluble P determination has also shown rather poor inter-laboratory agreement with variability ranging from 25.6% to 57.2%

c.v. throughout the cross-check exercise. The high inter-laboratory variability is attributable to the low values of soluble P in the soil samples as well as to the varied extracting solutions employed by the different laboratories. Some improvement was observed when a standard extracting solution of acid fluoride - 0.3 N ammonium fluoride at pH 1.8 was subsequently introduced for the cross-checks.

#### LEAF ANALYSIS

Leaf analysis has the similar components of errors as soil analysis. A cross-check on rubber and oil palm leaves carried out in 1964 (Middleton *et. al*) by 4 laboratories (RRIM, Socfin, Dunlop, Chemara) showed good inter-laboratory agreement in chemical analysis of leaves. The laboratory errors were completely swamped by field sampling errors if samples were taken on a tree-to-tree basis. However, the field sampling errors were considerably lessened and brought close to the laboratory ones if a sample represented a number of trees randomly taken in the same field (Table 6). The present sample size is 30 trees from a population of 3,600 trees.

#### Laboratory Errors in Leaf Analysis

The main components of laboratory error are again attributed to day, operator, digestion and sub-sampling. Middleton *et. al* (1966) has reported that the errors associated with these components are small both for major and minor elements in leaves of Hevea brasiliensis (Table 7).

Lancaster (1971) also obtained values of total error of similar magnitude for the major elements (N,P,K, Ca, Mg and Mn) using automated and atomic absorption spectrophotometric procedures. Reproducibility and recovery studies gave very good agreement. Average recovery was  $100 \pm 1\%$  while reproducibility was within 2%.

### Inter-Laboratory Cross-Checks

Routine inter-laboratory cross-checks were initiated in 1969. During the same period local laboratories also began participating in Round Robin Cross-Checks organised by the Laboratory of Wageningen, Holland. Table 8 gives information on the number of cross-checks carried out.

Table 9 gives the essentials of the analytical techniques employed by the different laboratories while Table 10 gives the results.

A comparison of inter-laboratory variabilities is attempted in Table 11.

On the whole there has been some improvement in the inter-laboratory agreement since its initial stages for the local cross-check exercise. Values for N, P, K show below 5% coefficient of variation, while Mg and Ca although still in the 5-10% variability range have somewhat lower c.v. values as compared to that in 1969. The significant cut down in the inter-laboratory variation may be due to the fact that most laboratories are now employing atomic absorption spectrophotometry for their analysis (Lim Han Kuo & Liew Kok Leong, 1978).

In the minor elements too, there has been some improvement. The occurrence of rogue results have nevertheless, inadvertently, contributed to the wide variation and poor inter-laboratory agreement of the analysis. 'Rogue results' are those which vary from the mean by  $\pm 10\%$  or more. They can be attributed to several factors such as contamination, calculation error, typing error or even general analytical error.

The agreement for Cu has improved somewhat but that of Zn has worsened. The high inter-laboratory variability is also due to the low concentrations of these elements in plants.

For the Wageningen cross-check exercise, the inter-laboratory agreement is less satisfactory. Somewhat higher c.v. % is observed for most of the elements as compared to that of the local cross-check. This may be due to the wide variety of plant materials which are used in these cross-checks e.g. salad, oats, barley, etc.

#### CONCLUSIONS

Studies have shown that both in soil and foliar analyses, laboratory analytical errors are very much smaller than field sampling errors. In the case of soil analysis, extraction procedures form the major component of laboratory error. In the case of plant analysis, the major elements do not present much problems in obtaining good precision. Standardization of analytical procedures has resulted in some improvement especially in the case of soil analysis and the analysis of minor elements in plant samples.

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Table 1. Soil sampling variability (% C.V.)<sup>\*</sup>

	Rengam	Malacca	Kuantan	Munchong	Klau	Serdang
pH	2.5	3.7	<u>1.9</u>	<u>5.7</u>	5.4	3.3
C%	13.8	13.3	<u>19.3</u>	<u>13.2</u>	16.6	18.8
N%	14.2	<u>11.5</u>	16.9	13.6	16.2	<u>21.0</u>
Total P	<u>10.3</u>	41.9	28.2	<u>45.6</u>	17.2	10.4
Acid-fluoride P	30.2	72.5	31.0	<u>167.7</u>	<u>24.0</u>	24.7
6 N HCl-K	46.6	27.7	<u>15.1</u>	49.1	<u>105.1</u>	68.2
6 N HCl-Ca	38.9	53.2	<u>25.3</u>	<u>106.9</u>	72.3	38.7
6 N HCl-Mg	38.5	25.0	29.0	<u>23.8</u>	47.5	<u>64.9</u>
Exch. K	<u>16.9</u>	25.1	24.9	25.9	<u>279.9</u>	23.6
Exch. Ca	<u>46.4</u>	68.6	78.3	73.3	<u>167.7</u>	49.7
Exch. Mg	24.8	71.6	<u>21.6</u>	22.9	<u>80.2</u>	52.2

Results are for 40 samples of 3 cores per sample (0-15 cm soil depth)  
 Minimum and maximum values are underlined

\* Ng et. al (1971a)

Table 2. Sample size (no. of random points) for precisions of 20%<sup>\*</sup>

	Rengam	Malacca	Kuantan	Munchong	Klau	Serdang
pH	1	1	1	1	1	1
C	2	2	4	2	3	4
N	3	2	3	2	3	5
Total P	2	18	9	22	3	2
Acid-fluoride P	10	54	10	290	6	7
Exch. K	3	7	7	7	800	6
Exch. Mg	7	53	5	6	66	28
Exch. Ca	22	48	63	56	288	26
6 N HCl-K	23	8	3	25	113	48
6 N HCl-Mg	16	7	9	6	24	44
6 N HCl-Ca	16	29	7	118	54	16

Results are for 3 core samples for 0-15 cm soil depth

\* Ng et. al (1971a)

Table 3. Total test error and its components (% C.V.)\* in soil analysis

	Day	Operator	Digestion	Duplicate Instruments	Total Error
pH	0.5	0.2	0.4	0.7	0.9
C	1.0	1.0	1.7	-	2.2
N	1.7	0.9	2.3	1.6	3.4
Acid-fluoride P	1.9	3.6	4.4	1.7	5.9
Total P	0	1.1	2.6	1.0	3.0
Exch. K	2.3	2.6	4.7	1.1	5.9
Exch. Mg	0	1.0	3.7	1.1	4.0
Exch. Ca	0	0	9.9	2.7	10.2
6 N HCl-K	0	6.0	7.2	1.5	9.5
6 N HCl-Mg	0	14.6	7.4	0.8	16.4
6 N HCl-Ca	2.4	4.2	11.5	1.2	12.5
Total Mn	2.1	5.0	3.1	2.4	6.7

\* Ng et. al (1971b)

Table 4. Soil cross-checks information

Cross-Checks	Samples	No. of Samples	Laboratories	Meetings
1966	DOA Sabah	10	DOA (Sabah)	Sabah 1967 (1st meeting)
	DOA Sarawak	10	DOA (Sarawak)	
	DOA, P. Malaysia	10	DOA (P. Malaysia)	
			RRIM	
	- do -		- do -	K.L. 1968 (2nd meeting)
1969	RRIM	6	Above 4 + OPRS	
1970	DOA Sarawak	6	- do -	
1971	DOA Sabah	6	- do -	
1972	DOA, P. Malaysia	6	Above 5 + Chemara and ARAB	K.L. 1973 (4th meeting)
1974	DOA Sarawak	6	Above 7	
1975	DOA Sabah	6	- do -	
1975	DOA, P. Malaysia	6	Above 7 + HRU and FELDA	
1976	DOA Sarawak		analysis suspended because of heat treatment during quarantine	
1977	RRIM Sarawak	6	Above 9	
			- do -	

Table 5. Average inter-laboratory C.V. % in soil analysis

Parameter	1969	1970	1971	1972	1974 (Sarawak soils)		1975 (Sabah soils)		1975 (P. Malaysia)	
	Overall mean	Average c.v. %	Overall mean	Average c.v. %	Overall mean	Average c.v. %	Overall mean	Average c.v. %	Overall mean	Average c.v. %
pH (H <sub>2</sub> O)	1.5	3.1	4.1	4.5	4.85	2.7	5.0	3.3	2.95	5.2
pH (0.01 N KC1)	3.8	3.0	3.1	2.7	4.17	2.3	4.4	4.1	2.83	4.2
Carbon %	25.6	12.5	9.6	11.9	2.03	3.2	1.32	12.8	4.27	10.9
Nitrogen %	11.1	12.0	24.2	10.0	0.185	6.5	0.15	11.5	0.158	10.4
Soluble P ppm	36.6	50.1	41.9	57.2	9.6	35.6	7.1	25.6	8.3	33.8
Total P ppm	-	-	-	53.0	201	11.4	242	19.5	165	10.9
C.E.C. m.e.%	15.6	32.8	20.2	31.0	11.6	17.3	7.76	29.7	21.66	20.3
Ex. Ca m.e.%	28.7	18.7	16.3	22.8	2.44	20.9	1.78	42.6	0.25	33.2
Ex. Mg m.e.%	23.0	17.8	22.6	27.0	1.24	14.0	0.67	15.5	0.39	29.1
Ex. K m.e. %	19.1	27.6	29.4	11.3	0.19	20.5	0.22	24.2	0.21	28.3
Ex. Na m.e.%	-	-	-	-	0.08	43.6	0.06	27.9	0.16	35.7
Conductivity mho .cm <sup>-1</sup>	-	-	-	23.9	36.7	16.8	57	23.0	722	10.6
Sulphate %	-	--	-	-	-	-	-	-	0.12	11.9
Chloride %	-	-	-	-	-	-	-	-	-	-
Clay %	23.3	30.9	14.4	5.1	26.3	16.4	27.5	17.0	41.0	16.4
Silt %	32.7	22.4	13.1	18.8	29.3	8.6	16.4	10.2	33.8	10.7
Fine sand %	17.2	14.2	9.0	16.2	31.1	23.1	42.0	15.6	21.4	30.3
Coarse sand %	15.5	21.7	16.0	9.9	11.0	52.3	12.0	28.6	2.0	87.8

Table 6. Dependence of field errors on sample size  
in foliar analysis for rubber

Element	Lab. errors	C.V. %	
		Field errors sample size : 1 tree	Field errors sample size : 20 trees
N	1.55	8.30	1.86
P	1.60	14.68	3.28
K	2.21	32.95	7.37
Ca	1.56	25.72	5.75
Mg	2.39	16.75	3.74
Mn	3.99	150.29	33.61

\* Middleton et. al (1964)

Table 7. Errors in leaf analysis\*

Parameter	% Coefficient of Variation					
	Mean %	Anal error	Sub-sampling	Day	Operator	Total error
N	3.32	0.92	0.51	1.41	0	1.76
P	0.23	0.44	1.32	0.35	0.86	1.68
K	1.17	0.98	0.14	0.94	0.56	1.48
Mg	0.25	1.15	1.07	1.27	2.44	3.17
Ca	0.57	0.95	1.14	0.93	2.92	3.41
Mn	157	0.74	2.91	0.81	0	3.11
S	0.24	2.47	5.20	6.37	-	8.58
ppm						
	Anal + sub-sampling					
Al	230		2.38			3.37
B	13.3		2.64			4.17
Cu	13.7		1.62			3.24
Fe	213		5.86			6.48
Mo	0.20		3.12			6.69
Zn	21.4		1.86			6.53

\* Middleton et. al (1966)

Table 8(a). Local foliar analysis cross-checks

Year of Cross-Check	No. of Samples	No. of cross-checks per year	Participating Laboratories
1979	6	2	OPRS, Unilever, DOA Sarawak, DOA P. Malaysia, Chemara, Dunlop, RRIM
1970	6	1	- do -
1971	6	1	- do -
1972	6	2	<u>Additional laboratories:</u> ARAB, Malayan Testing Lab., Pineapple Res. Centre, Agric. Res. Centre Sabah, Devon Processing Sdn. Bhd., Socfin Co. Bhd.
1975	6	3	<u>New additions</u> HRU, Uniroyal Mal. Plantations, FELDA, KL-Kepong, MARDI (minus Unilever)
1976	6	3	- do -
1977	6	3	- do -

Table 8(b). Wageningen foliar analysis cross-checks

Year of Cross-check	No. of Samples	No. of cross-checks per year	Participating Laboratories
1969	6	6	OPRS, Unilever, Chemara, Dunlop, RRIM, DOA P. Malaysia
1970	6	6	- do -
1971	6	6	<u>Addition:</u> MTL, Uniroyal
1972	6	6	- do -
1973	6	6	<u>Addition:</u> Socfin, HRU, DOA Sabah
1974	6	6	<u>Addition:</u> FELDA, MARDI
1975	6	6	<u>Addition:</u> ARAB, Devon Processing, KL-Kepong
1976	6	6	- do -
1977	6	6	- do -

Table 9. Methods used in the laboratories (1974)

Organisation	N	P	K	Ca	Mg	Mn	Fe	Cu	Zn	B
Uniroyal	D	C(VM)	F	EDTA	EDTA	C	C	C	n.d.	n.d.
Dunlop	C(Phenate)-A	C(VM)A	F	AA	AA	AA	AA	AA	AA	C
HRU	D	C	F	AA	AA	n.d.	n.d.	AA	n.d.	C(Carmine)
Felda	C(Phenate)-A	C(VM)A	F-A	AA	AA	n.d.	n.d.	n.d.	n.d.	n.d.
DOA Kuala Lumpur	C(Phenate)-A	C(VM)A	F-A	AA	AA or (EBT)-A	AA	C(Dipyridyl)-A	AA	AA	C(Carmine)
RRIM	C(Phenate)-A	C(VM)A	F-A	AA	C(EBT)-A	C(Formal-doxime)-A	AA	AA	AA	C(Carmine)
Socfin	D	C(VM)A	F	EDTA	C(Thiazole)	C(Peri-date)	C(Tiron)	C(Diethyl-dithio-Carbamate)	C(Zircon)	C(Azomethine-H)
MTL	D	C	F	AA	AA	AA	AA	AA	AA	C(Quinalizarin)
DOA Sarawak	C(Phenate)-A	C(VM)A	F-A	AA	AA	AA	AA	AA	AA	C(Carmine)
MARDI	D	C(VM)	F	EDTA	EDTA	n.d.	n.d.	n.d.	n.d.	n.d.
H & C OPRS	C(Phenate)-A	C(VM)-A	F-A	AA	AA	C(Formal-doxine)-A	C(Dipyridyl)	AA	AA	C(Carmine)
DOA Sabah	C(Phenate)-A	C(VM)-A	F-A	F or AA	C(EBT)	AA	AA	AA	AA	C(Curcumine)
Chemara	C(Phenate)-A	C(VM)-A	F	AA	AA	AA	AA	AA	AA	C(Carmine)
ARAB	D	C(VM)	F	AA	AA	C(Peridate)	AA	AA	AA	C(Carmine)
Devon	C(Phenate)-A	C(VM)-A	F-A	AA	AA	AA	AA	AA	AA	C(Carmine)

A = Autoanalyser

F = flame emission

AA = Atomic absorption

Phenate = Alkaline Phenate

C = Colorimetry

n.d. = not determined

D = Distillation &amp; titration

VM = Vanado-Molybdo

EDTA = Edta titration

Table 10(a). Average Interlaboratory C.V. % of local cross-checks

Parameter	Year						
	1969	1970	1971	1972	1975	1976	1977
N	5.0	4.2	3.3	2.6	6.1	4.5	4.0
P	5.8	3.4	4.3	8.3	3.1	3.7	4.0
K	5.9	5.7	7.2	7.9	8.5	5.3	3.3
Ca	7.9	10.8	11.3	8.9	11.8	8.2	7.4
Mg	8.6	10.3	7.2	10.5	15.0	6.6	8.3
Mn	9.3	13.4	9.2	12.2	13.8	11.0	15.0
Fe	15.9	16.2	35.2	13.1	12.0	6.3	5.1
Cu	27.0	32.0	12.7	30.0	32.6	18.6	11.7
Zn	19.4	27.8	13.6	23.4	30.7	26.9	27.2
B	10.0	9.6	11.7	20.8	17.6	18.6	6.0

- (i) Results for 1969-1971 taken from Singh 1971b
- (ii) Results for 1972 taken from Singh et. al 1972
- (iii) Results for 1973 and 1974 were not available as the local cross-check was stopped temporarily and reintroduced again in 1975
- (iv) Results for 1975-1977 taken from Lim Han Kuo & Liew Kok Leong 1978
- (v) Values are calculated after exclusion of all 'rogue' results.

Table 10(b). Average Interlaboratory C.V. % of Wageningen cross-checks

Parameter	Year					
	1969	1970	1971	1972	1973	1974
N	5.0	3.7	6.3	5.1	4.9	4.5
P	5.2	6.4	4.6	6.6	6.5	6.6
K	4.7	6.1	7.6	7.9	7.8	6.2
Ca	8.6	7.2	17.2	9.7	11.2	9.8
Mg	7.1	8.4	9.7	10.7	10.3	9.3
Mn	13.1	17.9	12.2	17.7	12.1	10.7
Fe	14.7	13.5	13.3	16.1	13.0	12.6
Cu	22.1	19.6	18.3	29.9	19.7	15.1
Zn	14.6	18.9	33.9	12.8	14.1	13.4
B	13.9	14.2	20.1	27.8	11.6	12.5

- (i) 1969-1972 results taken from report by Singh 1972
- (ii) 1973-1974 results taken from report by H.F. Yeoh. Meeting on Standardization of Plant Analysis, Chemara Research Station, Seremban
- (iii) Values are calculated after exclusion of all 'rogue' results.

Table 11. Comparison of inter-laboratory variabilities

% C.V.	Local Cross-Check		Wageningen Cross-Check	
	1969	1977	1969	1974
0 - 5	N	N, P, K	N, K	N
5 - 10	P, K, Ca, Mg, Mn, B	Fe, B, Ca, Mg	P, Mg, Ca	K, P, Mg, Ca
10 - 15	-	Cu, Mn	Mn, B, Zn, Fe	Mn, B, Fe.
Above 15	Fe, Zn, Cu	Zn	Cu	Cu

## ROUTINE DETERMINATIONS OF SOIL PHYSICAL PROPERTIES IN MALAYSIA

by

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### ABSTRACT

The need for soil physical measurements has been described by many authors. Crop growth and uptake of nutrients are greatly influenced by soil physical properties, particularly by the moisture status. Knowledge of soil physical properties is helpful in understanding pedological relationships better and can provide a physical qualification of morphological criteria used to differentiate homogeneous soil units.

The current methods of determination of soil physical parameters practised by major soil research organisations and institutions were evaluated and discussed.

### INTRODUCTION

The need for soil physical measurements has been described by many authors (Chan and Soong, 1971; Guha, 1971; Law and Paramanathan, 1971). Some of the important reasons are:

1. Uptake of nutrients are greatly influenced by soil physical properties particularly its moisture relationships.
2. Crop growth is very much dependent upon direct availability of moisture.

3. Knowledge on soil physical properties is helpful in understanding soil pedological relationships better and can provide physical qualification of morphological criteria used to differentiate homogenous soil units. For example, field textural judgement can be confirmed by the particle size distribution. Bulk densities could be used to differentiate the marrine alluvium soils (e.g. Selangor series) from the inland soils (e.g. Rengam series). Chan and Soong (1971) showed that Selangor series having bulk density about 0.97 gm.  $\text{cm}^{-3}$  can be differentiated from inland sedentary and inland alluvial soils which have bulk density values of 1.00 gm.  $\text{cm}^{-3}$ .

Soil physical properties that are commonly determined by many Malaysian laboratories are the particle size distribution, soil moisture characteristics, bulk density, particle density and soil moisture. This note briefly discusses the current methods of determinations of these soil physical parameters. The controversial points will be pointed out for further discussion.

#### PARTICLE SIZE DISTRIBUTION

The pipette method is commonly employed in many laboratories.

Pretreatment and Dispersion -  $\text{H}_2\text{O}_2$  (30%) is used to destroy the organic matter. In many tropical soils it has been found that addition of 0.2 N HCl is necessary to remove free iron oxides. For the dispersion, mixtures of sodium hexa-metaphosphate ( $\text{NaPO}_3 \cdot 6$ ) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) have proved particularly effective.

Sieving and sedimentation - International Society of Soil Science (ISSS) system of textural classification is used to separate the particle sizes by sieving and sedimentation. Sedimentation times for particles settling through water for a depth of 10 cm are

calculated at 28°C. Thus at Universiti Pertanian Malaysia (UPM) the sedimentation process is carried out in a constant waterbath at 28°C.

Pipette sampling apparatus - Special pipette with holder.

One common error made by many laboratories is when textural class of the soil is inferred using the textural triangle.

	<u>USDA (mm)</u>	<u>ISSS (mm)</u>
very coarse sand	2 - 1	-
course sand	1 - 0.5	2 - 0.02
medium sand	0.5 - 0.25	-
fine sand	0.25 - 0.1	0.2 - 0.02
very fine sand	0.1 - 0.05	-
silt	0.05 - 0.002	0.02 - 0.002
clay	0.002	0.002

In USDA system sand is classified as particles having size more than 0.05 mm whereas in ISSS it is more than 0.02 mm. Since the textural triangle is constructed using USDA system, an error will be incurred when the results of particle size analysis based on ISSS system are used to find the textural class.

#### SOIL MOISTURE CHARACTERISTICS

Preparation of sample - in determining the soil moisture characteristics, undisturbed samples should be used. The pore space system of the soil dominates water retention at low pressures. The dependence of pore space on bulk density and structure therefore emphasizes the need for undisturbed samples, particularly at wet end of the moisture characteristic curve. Undisturbed samples can be obtained using the core method. Bigger cores of 7.65 cm diameter are generally preferred over the smaller cores of 5.00 cm diameter to minimize the sampling disturbance.

In some laboratories in Malaysia, sieved samples (1-2 mm) are quite often used in soil moisture characteristic measurements because such samples are easy to take. These measurements have been correlated quite well with other soil water measurements. However, the usefulness of these correlation should be carefully studied before any interpretations are made especially in the low pressure ranges where the soil water regime is structure dependent.

Toward the end of the moisture characteristic curve water retention is governed by the specific surface of the soil. The state of the samples (disturbed or undisturbed) is therefore of relatively little concern at pressures above 1 bar.

Measurements - the normal pressure ranges used in determining the moisture characteristics curve are 0.001, 0.01, 0.1, 0.33, 1, 3 and 15 bars. Except for the 0.001 bar pressure, the pressure plate apparatus are used. It is quite often the results are erratic at low pressure ranges (0.1 - 1 bar) when using this apparatus. Thus the use of hanging water column is preferred. From practical point of view the moisture characteristic at pressure about 15 bars are less important.

To reach equilibrium the samples must be kept for a certain period depending on the type of samples and type of soils:

undisturbed samples - 4 - 7 days and

disturbed samples - 2 - 3 days (Erh K.T., personal communication).

Different laboratories use different source of pressure. At Malaysian Agricultural Research and Development Institute (MARDI) a compression pump is used. At UPM, compressed Nitrogen gas is used. Although it is cheaper, compressed air is not used because it contains some moisture.

## BULK DENSITY

Dependent of bulk density on pore space and soil moisture indicating the necessity of using undisturbed samples. In soil moisture characteristics determination the bulk density values are used to convert the moisture contents from weight to volume basis. Thus when using sieved samples for the determination of soil moisture characteristics, the results can only be expressed on weight basis.

Preparation of sample - undisturbed soil core is taken in the field using a core sampler. At UPM cores of 7.65 cm diameter are used. In other laboratories smaller cores of 5.00 cm diameter are instead used. For proper design of the core the area ratio (A) should be 0.1 for ideal lateral compaction.

$$A = \frac{(\text{external diameter})^2 - (\text{internal diameter})^2}{(\text{internal diameter})^2}$$

For example, the UPM cores:

$$A = \frac{7.95^2 - 7.65^2}{7.65^2} = 0.007 \approx 0.1$$

Precautions - bulk density samples should not be taken under extreme wet or dry conditions. Sampling during very wet condition may cause compression of the samples and if during very dry condition may result in shattering of the cores.

Alternate method - if it is not possible to use core samples i.e. stony soils or very compact soils, the bulk density can be determined by using paraffin waxed-clod method.

## PARTICLE DENSITY

The particle density of soils can be easily determined using specific gravity bottle. Only disturbed sample is needed and the procedures are fairly straight forward.

## PORE SPACE

In calculating total pore space (total porosity), the following formula may be used:

$$P = \frac{(D_s - D_b)}{D_s} \times 100$$

where:  $P$  = Total porosity  
 $D_s$  = Particle density  
 $D_b$  = Bulk density

## SOIL MOISTURE DETERMINATION

The simplest and most accurate method for soil moisture determination is the gravimetric method. The samples are heated at  $105^{\circ}\text{C}$  for 24 hours. At UPM for rapid soil moisture determination, a semi automatic moisture oven is used. Exactly 10 grams of sample is placed in the oven for approximately one hour. The percent moisture by weight can be read from a build-in balance.

The measurement of soil moisture for water balance studies *in situ* is being carried out at UPM using neutron moisture meters. At present we are still in the process of calibrating them for two UPM soils.

## SOIL WATER POTENTIAL

The soil water potential can be measured *in situ* using tensiometers. The factory - manufactured tensiometers with pressure gauges are available. However, the cups are usually of big sizes and therefore are less sensitive to potential changes.

The limitation of using tensiometer for measuring soil water potential is that it only works in the low potential ranges

i.e. less than 0.8 bar. However, it can be used perfectly to measure soil water potential at greater depth where the potential seldom falls below 0.8 bar.

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## MEASUREMENT OF SOIL NUTRIENT AVAILABILITY IN MALAYSIA

by

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## ABSTRACT

Soil nutrient availability could be conveniently determined by extracting the nutrients from the soils by solution of acids or salts. However, in recent years, with better knowledge on ion-exchange reactions in soils, the approach to assessing soil nutrient availability has somewhat changed. In this present paper the use of thermodynamic indices (quantity/intensity) and a strong cation exchange resin for determining soil nutrient status and plant performance will be examined with particular reference to Hevea.

## INTRODUCTION

Many studies have been conducted to determine that portion of soil N, P, K, Ca or Mg which is considered to be available to plants. In Malaysia, routine laboratory methods for assessing soil nutrient availability were based on extraction procedures using mostly acids of different strengths or salt solutions. The soil nutrient status was assessed by the Kjeldahl digestion method for N, concentrated  $H_2SO_4/HClO_4$  and  $NH_4F/HCl$  extraction methods for total and easily soluble P, 6 N HCl and  $NH_4OAc$  extractions for total and exchangeable K, Ca and Mg. Although these methods have been found to be satisfactory for the various soil types under different field conditions, they have certain shortcomings in that they are unable to discriminate soils with low to very low nutrient levels.

In recent years, with better knowledge on ion-exchange reactions in soils, the approach to assessing soil nutrient availability has somewhat changed. The present paper gives an account of recent development in soil chemical methods for assessing soil nutrient availability. In addition, use of clay mineralogical properties and surface areas of clay fractions which could assist in the determination of the nutrient supplying power of the soils were also briefly mentioned.

#### DISCUSSION

In the early 1970s, the thermodynamic quantity/intensity (Q/I) relationship was developed to determine not only the amount of available nutrients in the soil, but also the ease with which these nutrients can be removed from the soils (Singh, 1970 and 1971; Singh and Talibudeen, 1969). Subsequent work had led to the use of a strong cation exchange resin for assessing the amount of exchangeable and non-exchangeable cations (Lau, 1975). The application of these methods to determine soil nutrient status and plant performance, particularly Hevea, shall be separately examined.

#### Quantity/Intensity Relationship

The quantity/intensity relationship (for  $K^+$ ,  $Mg^{2+}$  and  $PO_4^{3-}$ ) of some Malaysian soils was first studied by Singh (1970, 1971) and Singh and Talibudeen (1969). The Q/I relationship of a soil for a cation like  $K^+$  relates the change in the amount of exchangeable  $K^+$  in the soil to the K intensity index which is the activity ratio,  $a_K/a_{Al}^3$  of the equilibrium soil solution. The intensity index gives the strength with which the nutrient is held in the soil while the term 'quantity' denotes the amount of the ion present at a definite intensity in the soil. The rate of change of quantity with intensity which is obtained from the slope of the Q/I curve determines the buffering capacity of the soil and this index measures the capacity of the soil to buffer any increase or decrease of the

nutrient ion content in the soil. In the Q/I relationship,  $Al^{3+}$  is taken as the reference ion since this ion is always present in competition with  $K^+$  and  $Mg^{2+}$  cations in the acid Malaysian soils.

Several investigations were initiated to test the usefulness of these thermodynamic indices over the conventional indices of acid-extractable and exchangeable values for K in relating uptake and growth of plants. Singh and Talibudeen (1969) showed that the potassium buffering-capacity value obtained from the Q/I relationship was better than the acid-extractable and exchangeable values for K in relating uptake by Pueraria in a glasshouse experiment (Table 1). For phosphorus, both conventional and thermodynamic indices relate to levels of phosphate manuring as well as greenhouse cropping. Additional work by Singh (1971) showed that the thermodynamic indices were more sensitive than the conventional soil indices in reflecting both past and current manuring. The order of sensitivity quantity exchangeable acid-extractable buffering capacity.

Although the method based on thermodynamic concepts appeared to be promising for determining soil nutrient status, Lau et.al. (1972) found that this method was not much superior than the conventional method for assessing nutrient status of soils under Hevea (Table 2). Exchangeable, acid-extractable, quantity and intensity values for K appeared to correlate with leaf-K while buffering capacity values did not relate to any of the plant indices (leaf-K, growth and yield of latex).

#### Soil K and Mg Status Using a Cation-Exchange Resin

Extraction of K and Mg from five representative Malaysian soils by a  $H^+$ -saturated cation-exchange resin showed that both exchangeable K and Mg were rapidly removed by the resin and that the soils contained variable amounts of non-exchangeable K and Mg, depending on the clay mineralogical properties (Lau, 1975, 1978). Besides K and Mg, large amount of Al was also extracted. The large amount of Al which varies from 33.5 to  $8.3 \text{ me.}100 \text{ g}^{-1}$  soil suggested that

Al is competing with K and Mg for exchange sites in the resin. Additionally, this also implied that Al plays an important role in affecting uptake of K and Mg by plant roots which is simulated by the cation-exchange resin.

The extent to which uptake of K by Hevea (measured by leaf-K values) is related to the soil K status determined by the  $H^+$ -saturated cation exchange resin and the various conventional methods are presented in Table 3. Correlations between K extracted by the resin and K uptake by the plants are good. Compared with acid-extractable K extracted by 6 N HCl and exchangeable K by N ammonium acetate, K extracted by  $H^+$ -resin provides a better assessment of soil K status with respect to uptake by Hevea.

In conjunction with the above study, rate studies on the adsorption and release of K and Mg by the soil to  $H^+$ -resin suggested that the fertility of the five soils investigated is controlled by several factors. Among the various factors are the clay mineralogical contents, organic matter and the specific surface areas of the exchange complex. The Selangor and Durian Series soils which contained appreciable amount of 2:1 type minerals released more K and Mg but at slower rates compared with the inland soils of the Kuantan, Munchong and Rengam Series soils. In adsorption studies, the inland soils reached saturation point at a faster rate than the coastal alluvial clays of the Selangor Series, suggesting that leaching losses would be appreciable in the former soils. Qualitatively, the reactivity of the clays could be related to the charge per unit area of the clays. The Selangor and Durian clays have the largest charge/unit area and thus would be able to retain nutrients more effectively.

#### CONCLUSION

Although soil nutrient availability could be conveniently determined by extracting the nutrients from the soils by solutions of acids or salts, factors controlling nutrient release and adsorption by the soil complex require further investigation. It is the

understanding of these factors that a fuller appraisal of the availability can be concluded.

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Table 1

Linear Correlation Coefficients of Soil Potassium Indices With  
Greenhouse Cropping by Pueraria

Soil K Index	Correlation coefficient(r)
	All 8 soils
Acid-extractable	0.95***
Exchangeable	0.83*
Quantity ( $Q_o$ )	0.83*
Intensity ( $I_o$ )	-0.36 NS
Buffer capacity (laboratory)	0.93***
Buffer capacity (cropping)	0.95***
Buffer capacity (exchangeable)	0.88**

\*\*\* P &lt; 0.001

\*\* P &lt; 0.01

\* P &lt; 0.05

NS: Not significant

After Singh and Talibudeen (1969)

Table 2. Correlation coefficient ( $r$ ) of soil-K indices with plant indices in Hevea

Soil K Index	Correlation Coefficient ( $r$ )								
	Leaf K (%)			Girth Increment (cm)			Yield (g/tree) of dry rubber		
	1	2	3	1	2	3	1	2	3
Acid-extractable (6 N HCl)	0.52**	0.19	0.47*	0.31	0.18	0.26	0.30	0.21	0.38*
Exchangeable (1 N $\text{NH}_4\text{OAc}$ )	0.48*	0.40*	0.43*	0.26	0.42*	0.15	0.21	0.38*	0.43*
Quantity ( $Q_o$ )	0.50*	0.44*	0.45*	0.25	0.43*	0.12	0.25	0.32	0.48*
Intensity ( $I_o$ )	0.42*	0.44*	0.21	0.28	0.20	0.15	0.20	0.16	0.21
Buffering Capacity (B.C.)	0.03	0.10	0.08	0.01	0.16	0.17	0.17	0.15	0.04

\*\*\* P < 0.001

\*\* P < 0.01

\* P < 0.05

Note: 1, 2, 3, refers to Experiment No.

After Lau et. al (1972)

Table 3

Correlation Coefficient of Soil-Potassium Indices With  
Leaf-Potassium of Hevea

Soil-potassium index	Correlation coefficient (r)		
	Experiment SE.104	Experiment SE.53	Experiment SE.29/2
Resin K	0.40**	0.45**	0.40*
Acid-extractable K (6 N HCl)	0.07	0.47**	0.52**
Exchangeable K (1 N $\text{NH}_4\text{OAc}$ )	0.36*	0.43*	0.48*
Number of samples	45	32	25

\* P &lt; 0.05

\*\* P &lt; 0.01

After Lau (1975)

## DISCUSSION

## SESSION 1

## 1. Soil chemical analysis in Malaysian Laboratories.

Mr. Mohd. Khanif Yusop enquired whether any significant difference is observed in the determination of exchangeable cation and cation exchange capacity between leaching and shaking methods. Mr. Sharifuddin Hj. Ab. Hamid replied that there may be some differences but further research is needed to quantify these differences. Dr. Mohinder Singh said that past studies have shown that both methods give similar results if properly carried out. He noted that the existing methods of determining cation exchange capacity do not necessarily take into account the nature of exchange sites of our highly weathered soils and more research towards this end are needed.

Dr. S. Paramanathan asked whether any of the laboratories mentioned determines Al and which method is employed. Mr. Sharifuddin Hj. Abd. Hamid replied that only RRIM does Al determination on routine basis. 1 N KCl is used with 1:5 soil to solution ratio and a few seconds shaking time. Al in the filterate is then determined by aluminon method. Mr. Poon Yew Chin commented that the determination of available Al has often been difficult. He said that if it is assumed that Al availability is related to Al solubility, then soil pH is the overriding factor. Hence, the exact soil pH in situ must be known in order to predict the amount of soluble Al.

## 2. Methods of leaf analysis in Malaysian Laboratories.

Mr. K. Sivanadyan enquired whether any difference in N analytical values exist between the Kjeldahl and Colorimetric methods. Mr. Poon Yew Chin replied that very little difference may exist in N analytical values between the two methods. He stressed that the

errors in the analysis of nitrogen is associated mainly in the digestion methods used.

### 3. Precision of soil and leaf analyses in Malaysian Laboratories.

Dr. E. Pushparajah asked whether the Walkey and Black's method of determining percent carbon is applicable to acid sulphate soils. Dr. Mohinder Singh replied the method has been developed mainly for normal soils and may not be necessarily valid for problem soils. He further said that more attention should be paid to evaluate test methods for such soils.

To a question on what precision in field sampling and laboratory analysis should be used for fertilizer recommendation by Dr. E. Pushparajah, Dr. Mohinder Singh replied that based on current knowledge the existing precision may be acceptable but with increasing fertilizer cost, any further refinement will be welcome. He also noted that for improving fertilizer assessment, more attention should be given to field sampling techniques as they give significantly larger errors than the laboratory analytical techniques.

### 4. Measurement of soil nutrient availability in Malaysia.

Mr. Sharifuddin Hj. Abd. Hamid enquired whether the acid extractable cations are also highly correlated to plant nutrient content for the annual crops as in the case of rubber. Dr. Mohinder Singh commented that for annual crops, the thermodynamic parameters may be more meaningful as the plant has a large root system. Also in the tropics, there is a rapid release of nutrients from litter and soil matrix.

Mr. Mohinder also made the following comments:

1. In the case of macro differences between soils, any index is suitable but an index closer to the total reserves in soils is the desired index. On the other hand when looking at the micro

differences between soils with similar nutrient status, parameters like acid extractable cations are insensitive. Parameters which relate to the soils "energy" status are instead more likely indicators. The problem is that suitable methods to measure this in terms of actual field conditions are yet to be developed.

2. Cation exchange resins measure not only the nutrients which are more readily available but also some of the not so readily available ones. Hence they can be expected to reflect the soil thermodynamic status as well.

## **SESSION 2**

### **USE OF SOIL AND LEAF ANALYTICAL DATA IN MALAYSIA.**

PROBLEMS IN THE INTERPRETATION OF LEAF AND SOIL  
ANALYTICAL DATA FOR HEVEA MANURING

by

K. Sivanadyan  
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ABSTRACT

The paper outlines the difficulties an agronomist encounters when computing fertilizers discriminately for Hevea. Soil or leaf analysis by its own would be unsatisfactory for fertilizer recommendation. The philosophy of integrating soil and leaf parameters together with agronomic parameters have been discussed.

Although the art of interpreting soil and leaf analytical data can be problematic, yet a judicious approach to manuring is of paramount importance, particularly when costs of agro-management inputs are on the upward trend.

INTRODUCTION

The concept of discriminatory fertilizer application to Hevea trees, so as to suit conditions in specific areas, was attempted as early as in 1936 (RRIM, 1936). However, until recently, a rather general schedule of fertilizer for both mature and immature situations was still operative (Pushparajah, 1977). This approach of applying fertilizers discriminately is mainly based on the nutritional status of the trees and nutrient levels in the soil (Chan, 1972).

Chapman (1941) considered that the absolute amount of each nutrient in the leaf determines the productivity of any known stand.

After comparing between leaf and bark, Bolle-Jones (1957) inferred that leaves were superior to bark tissue as a sensitive indicator of nutritional status of trees. This was subsequently confirmed by others (Shorrocks, 1961, 1962a, b and c; Shorrocks and Ratnasingam, 1962). In order to provide general guidance for the assessment of leaf nutrient values the Rubber Research Institute of Malaya (1963) forwarded some critical leaf values for the major nutrient elements N, P, K and Mg (Table 1). For some time, the results of leaf analysis interpolated with Table 1, formed the basis of diagnosis of fertilizer requirements of Hevea, particularly for mature rubber.

Subsequent work (Pushparajah & Guha, 1968) showed that the response to fertilizers varied with not only the leaf nutrient content, but also the soil type and the nutrient content in the soil.

Table 1. 'Critical' leaf nutrient contents of Hevea  
(expressed as percentage of oven-dry sample)

Nutrient	Nutrient level below which response likely		Nutrient level above which response unlikely	
	Leaves exposed to sunlight	Leaves in shade of canopy	Leaves exposed to sunlight	Leaves in shade of canopy
Nitrogen	3.20	3.30	3.60	3.70
Phosphorus	0.19	0.21	0.25	0.27
Potassium	1.00	1.30	1.40	1.50
Magnesium	0.23	0.25	-	0.28

Further it was shown (Pushparajah & Chellapah, 1969) that the past and current cover management history would influence the fertilizer requirement and the response to fertilizers. For instance, the adoption of a pure legume cover policy during the immature phase would

result in large organic matter and nitrogen turnover into the soil. Such high soil N levels would eventually be available to the mature trees.

Ideally, the best approach would be one whereby a large number of experiments are set up to cover a large representation of the rubber growing areas in the country, but this is not feasible. This made it necessary to consider interpolation of results of limited number of experiments for wider application in the field, whereby the experimental data were related to a number of agronomic parameters such as soil series, soil and leaf nutrient contents, manorial history, ground cover conditions and clonal characteristics. The basic concept of this approach is integration of soil survey and soil and leaf nutrient data with results of fertilizer experiments to increase reproducibility for wider field applications. Thus, no single factor can stand on its own. Details of this philosophy and approach have been described by Chan (1972).

This paper discusses problems governing the interpretation of leaf and soil analytical data for discriminatory fertilizers recommendations in Hevea. A quantitative approach to such recommendation is also briefly discussed.

#### LEAF ANALYSIS

The technique for leaf sampling follows that recommended by Shorrocks (1964). The analytical values obtained only suggest the possible nutritional status of the trees at any one particular period or situation. These values are however, influenced by a multitude of factors, thus complicating the interpretation of data. Major nutrient contents can be affected by the following:

## Environmental influence

## (a) Soil factors

The existence of a rather positive relationship between soil and leaf nutrient status in mature rubber was demonstrated by Guha and Yeow (1966). This relationship was, however, recognised in commercial situations during the period (early 1960's), where fertilizers application to mature Hevea was restricted. Nevertheless, such information though obtained from survey data was also confirmed in experimental areas (Pushparajah and Guha, 1968). In a more recent investigation in smallholdings by Pushparajah et. al. (1972), the positive influence of soil variations of leaf nutrient levels was again recognised.

## (b) Seasons

It is inevitable that the sampling period or date can influence leaf nutrient contents. During the year monthly variations in leaf levels arise, as shown by Beaufils (1955). The author ascribed such monthly variations to the vegetative cycle or leaf age.

Besides time and date of sampling, another factor that can influence leaf nutrient status is the season or weather. This variation is observed to be independent of other agronomic variables. In an investigation conducted over seven years, the ranges in various leaf nutrient levels obtained are shown in Table 2. All samplings were carried out when leaf age was about 120 days of leaf emergence.

Large variations in leaf nutrient contents, beyond the limits of sufficiency and deficiency, were observed. These wide variations, resulting from seasonal effects, are expected to affect interpretation of the nutritional status of areas as determined by leaf analysis. In view of such uncertainties, Pushparajah (1973) recommends the monitoring of yearly variations in leaf nutrient contents from control plots and using these seasonal fluctuations for adjusting actual leaf values.

Table 2. Seasonal variation in leaf nutrient content in control plots of manuring experiment (1964-1970)

Soil Series	Clone (age)	Nutrient content, %			
		N	P	K	Mg
Malacca	BP 86 (1951)	3.05-3.33	0.22-0.26	0.96-1.34	-
Serdang	PB 5/51 (1962)	2.98-3.40	-	1.53-1.91	0.26-0.34
Rengam	GG 1 (1957)	3.06-3.58	-	1.06-1.44	0.19-0.29
Jerangau	LCB 1320 (1958)	-	0.17-0.25	0.52-0.88	-
Selangor	Tjir 1 (1954)	3.43-3.72	0.23-0.27	1.19-1.49	0.28-0.33

#### Agronomic-management influence

##### (a) Ground cover policy

The type of ground cover maintained during the immature phase exerts a considerable influence on the nutritional status of trees, particularly nitrogen and to some extent potassium (Watson et.al. 1964). Studies by Pushparajah and Chellapah (1969) besides confirming this effect also showed that cover condition could affect P contents of leaves. Particularly, legume covers were able to enhance leaf P levels even in the absence of phosphate applications. The implication of this enhancement is the possible ability of legumes to cycle phosphates from lower soil depths in a readily available organic form.

Similarly during the mature phase the type of covers will dictate the nutrition of trees, particularly that for nitrogen. For instance the leaf N levels in mature stands differed in relation to the underlying covers according to bare (3.63%), *Ottochloa nodosa* (grass) (3.62%) and *Nephrolepis* sp. (fern) (3.47%), suggesting the undesirability of a vigorous and dense fern cover.

## (b) Fertilizer applications

Fertilizer applications can also influence leaf nutrient levels and thus confound interpretation of analytical data. Leaf N, P, K and Mg levels will positively increase when these fertilizers are applied (Shorrocks, 1961). Application of nitrogen can result in a suppression leaf K and Ca in low K soils but in high K soils the reserve effect tends to hold (Pushparajah, 1973). The variation influence of fertilizers on leaf nutrient contents can be summarised, as given in Table 3.

Table 3. Effect <sup>(a)</sup> of fertilizers on leaf nutrient content

Fertilizers	% N	% P	% K	% Mg	% Ca
Ammonium sulphate	+	0/+	-/+	0/-/+	-
Rock phosphate	0/+	+	0	0/+	+
Muriate of potash	0/+	0	+	-	-
Kieserite	0	0	-	+	0

Note: (a) 0 = no effect; - = depressive effect;  
+ = positive effect

The net effect of fertilizers can either be synergistic or antagonistic and hence when considering fertilizer needs the soil levels must also be considered.

## (c) Time of fertilizer application

The time of application of fertilizers in relation to wintering also influences the nutrient levels in leaves. Significant uptake was observed when fertilizers were applied within four months of refoliation. But apparently no uptake of nitrogen was observed when the nitrogenous fertilizers were applied at six months after

refoliation. Sampling in relation to time interval after fertilizer application would also influence nutrient contents and hence if the fertilizer history is not known, then interpretation of leaf analysis can give misleading results.

#### Factors inherent to the tree

##### (a) Type of planting material

Especially for nitrogen and potassium the leaf nutrient content is dependent on the type of clone or planting material (Pushparajah and Tan, 1972). As an example, the potassium status of PB 86 and PB 5/51 in control plots of experiments ranged between 1.5% to 1.8%. Yet responses to potassium applications resulted in these clones, although the "critical" range (Table 1) was between 1.3% to 1.5%. In clone GT 1, with control leaf N ranging between 3.5% to 3.7%, yield response to nitrogen applications were evident. This indicates the need to consider clonal characteristics when using leaf analysis as a diagnostic criteria.

##### (b) Rootstock influence

Both greenhouse and field investigations have demonstrated the major influence of root stocks on leaf nutrient levels. In the main LCB 1320, rootstocks tend to sustain inherently phosphorus and potassium levels in leaves. However, plants with rootstocks of clones Tjir 1 and PB 5/51 invariably always exhibited higher leaf nutrient levels than other materials like RRIM 600 rootstocks.

##### (c) Crown influence

In crown budded trees, the type of crown clone has been reported to dictate the observed leaf nutrient levels (Pushparajah and Tan, 1972). These workers showed that the crown of RRIM 612 on RRIM 600 trunk gave the lowest potassium content in leaves when compared to other crowns. This reflected the characteristics of RRIM

612 which tends to show a lower uptake ability for potassium. This crown had a vast influence on RRIM 600 trunk which on the contrary as a crown component would lead to higher potassium content in the leaves.

#### Position of sampled leaf

The influence of position of leaf is well known. For use as a diagnostic criterion leaves to be sampled can be confined to a particular position. For immature rubber, leaves exposed to the sun are sampled while in mature trees, low shade leaves can be used.

#### Influence of exploitation system yield

With similar soil and cover management, different exploitation methods, resulting in differential yields, lead to differences in leaf nutrient contents (Pushparajah et. al. 1972). This is indicated in the following data:

Treatment	Yield (kg.ha <sup>-1</sup> .yr <sup>-1</sup> )	Leaf nutrient content (% dry wt.)		
		N	K	Mg
Unstimulated	1390	3.55	1.44	0.22
Ethrel - 10%	2570	3.40	1.26	0.21

Sivanadyan et. al (1972) have shown that the amount of fertilizers applied for a given clone and age would be dictated by the yield and the exploitation method. Hence, manuring in the absence of the knowledge of yield and based solely on the leaf nutrient criteria, would lead to temporary nutrient deficiencies due to higher drainage.

## Adjustments for leaf analysis values

Guha & Narayanan (1969) had shown that there was a variation in leaf N and K contents with leaf age, with both tending to decrease with increase in age. In addition, they showed that there was a relation between leaf Ca and age of leaves exposed to light. Extension of this work to leaves in the shade (Pushparajah & Tan, 1972) showed that similar relationships existed. Based on this work, correction factors for leaf age have been introduced. The corrections are calculated to be as follows:

- (i) For areas (a) high in calcium or (b) medium in calcium and/or receiving normal calcium in phosphatic fertilizers:
  - (a) when leaf Ca is higher than 0.6% for common clones except RRIM 501 and PB 107, increase observed in leaf N values by 0.087% and observed in K content by 0.052% for every 0.1% by which the Ca value is above 0.8%.
  - (b) increase leaf N and leaf K values observed at the level indicated in (a) for 0.1% by which the Ca value is above 0.6%. For RRIM 501 and PB 107, Ca of optimum age would be 0.4% and 0.5% respectively.
- (ii) For areas low in Ca and/or not receiving Ca containing fertilizers, increase in the observed values of leaf N and K by 0.19% and 0.67% respectively, for every 0.1% by which the observed Ca is higher than 0.6%.

In the interpolation of leaf nutrient levels to determine the deficiency and sufficiency of nutrients, the observed leaf values have to be adjusted using the correction values given above and then compared with the critical values. For such a critical value, a single standard would not be satisfactory for all clones. Further, the data available are insufficient for clearly defining the critical

values. Hence, a range of values was considered more appropriate. The range of values as proposed by Pushparajah & Tan (1972) are given in Table 4.

Table 4. Range of nutrient content in leaves at optimum age in the shade of canopy

Nutrient	Group	Low	Medium	High	Very high
N, %	I	3.20	3.12-3.50	3.51-3.70	3.71
	II	3.30	3.31-3.70	3.71-3.90	3.91
	III	2.90	2.91-3.20	3.21-3.40	3.41
K, %	I	1.25	1.26-1.50	1.51-1.65	1.66
	II	1.35	1.36-1.65	1.66-1.85	1.86
P, %	-	0.19	0.20-0.25	0.26-0.27	0.28
Mg, %	-	0.20	0.21-0.25	0.26-0.29	0.30
Mn, ppm	-	45	45-150	151	-

The classes were low, medium and high which are relative to the desired optimum status and the classifications are:

Low - levels are well below sub-optimal tending to visual deficiencies.

Medium - levels are sub-optimal.

High - levels can be considered luxury levels.

Very high - levels can be considered above luxury levels.

Besides the above range classes, tentative clonal groupings have been formed.

For nitrogen, they are: group I - clones RRIM 600, GT 1; group II - all clones except those in group I and group III; and group III - clones susceptible to trunk snap and branch break, e.g. RRIM 501, RRIM 513, RRIM 605, RRIM 623, etc. It is emphasised here that nutritionally, the values of leaf N of all classes in this group III could be considered low. Nevertheless these levels are a compromise between optimum mean and the reduction in susceptibility to wind damage heavy canopy.

For potassium, initially only two groups are proposed. They are: group I - RRIM 600, GT 1, PB 5/51 and PB 86; group II - all other clones.

The above levels for any element are applicable only when the levels of the other nutrients are satisfactory.

## DISCUSSION

### Leaf analysis

The foregoing discussions demonstrate that leaf analysis on its own would be unsatisfactory for discriminatory fertilizer recommendation. The philosophy of integrating soil and leaf parameters together with agronomic parameters have been discussed by Chan (1972). The relationships of leaf nutrient content to performance of rubber, variable though it may be, has been in use for quite some time and often reported. However, the relationship of soil to the performance of rubber have not been discussed in detail.

Owen (1953) showed the relationship of ammonium fluoride extractable P to the P requirements of rubber, while Guha (1963) showed that the total K status of the soil was related to the leaf K status of Hevea. Subsequently, Tan (1972) showed that for nitrogen total soil N and the C/N ratio of the 0-15 cm depth of the soil correlated well with leaf indices for rubber, while Lau et. al (1972) showed that good correlations with extractable K and reasonable

correlations with total K were obtained with both leaf K and yield of rubber. Lau *et. al* (1973) have shown that the ammonium fluoride extractable P as suggested earlier by Owen (1953) was related to leaf P contents and yield of rubber. These therefore imply that the leaf nutrient contents generally are a reflection of the nutrient content of the soil, the nutrient either being inherently present in the soil or artificially added as inorganic fertilizers. Further, these show that both leaf and soil nutrient contents correlate with yield.

The main problems that can be encountered when interpreting soil analytical data involve the analytical procedure itself.

In the case of soil P analysis, leaf P contents correlated well with soil values obtained by employing various extractants (Table 5). Among these the better extraction procedures were perchloric/sulphuric acid digestion, ammonium fluoride/HCl, hot and cold 0.1 N NaOH.

Table 5. Correlation of leaf P content with the various soil P indices

Extractions for Soil P	Correlation Coefficient (r)					
	SE.29/2	SE.62	SE.1/10	SE.36/2	SE.60	SE.53
Sulphuric/perch- loric extractable	0.63**	0.58**	0.65**	0.35*	0.45**	0.05
NH <sub>4</sub> F/HCl	0.38*	0.57**	0.59**	0.34*	0.53**	0.09
0.1 N NaOH (hot)	0.54**	0.67***	0.49**	0.33*	0.07	0.60***
0.1 N NaOH (cold)	0.37*	0.60***	0.55**	0.33*	0.57***	0.04
0.5 N NaHCO <sub>3</sub>	0.26	0.59**	0.58**	0.27	0.63***	0.01
0.5 N H <sub>2</sub> SO <sub>4</sub>	0.40*	0.57**	0.58**	0.34*	0.11	0.54**

\* significant at  $P < 5\%$  \*\* significant at  $P < 1\%$  \*\*\* significant at  $P < 0.01\%$

Thus it is rather essential to be aware of the type of extraction procedure one pursues when interpreting and comparing soil analytical values. The same holds for either potassium and magnesium analyses.

Another aspect to be borne in mind will be knowledge of the past agronomic history of the area studied. For instance if fertilizer application had been affected previously then soil sampling must avoid sites where fertilizers had been directed.

#### Discriminatory fertilizer recommendation

Owing to the vagaries in leaf and soil analytical data, a detailed knowledge of the field must also be known before fertilizer recommendation is made. Nevertheless both leaf and soil analyses constitute the main diagnostic criteria for assessing nutritional status of a stand of particularly mature rubber. For young rubber trees, their fertilizer needs are met through a preformulated programme, based on experimental evidence. In such situations leaf analysis is mainly used to serve as a guide to ascertain deficiency, imbalances or excess of nutrients.

For mature trees the interpretation of soil and leaf analytical data and the evaluation of nutrient needs have been subjected to variations. This has been due to varying experiences of individuals. In order to standardise fertilizer recommendations for any given situation, Pushparajah (1977) on the basis of experimental evidence, forwarded a quantitative approach. This is comprehensively illustrated in Figure 1 for nitrogen and potassium applications and Figure 2 for phosphate and magnesium applications.

#### CONCLUSION

The current discussion clearly outlines the difficulties an agronomist encounters when computing fertilizers discriminately for Hevea. Although the art of interpreting soil and leaf analytical

data can be problematic, for reasons discussed earlier, yet a judicious approach to manuring is of paramount importance, particularly when costs of agro-management inputs are on the upward trend.

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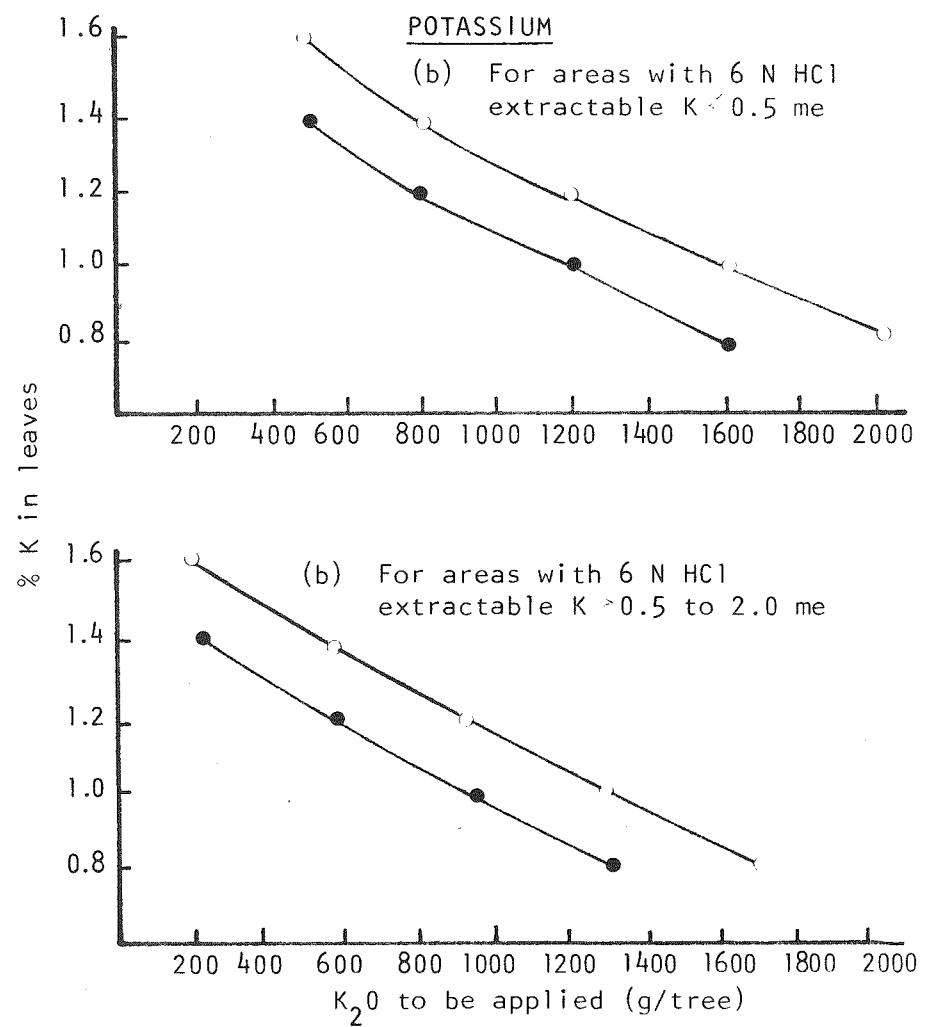
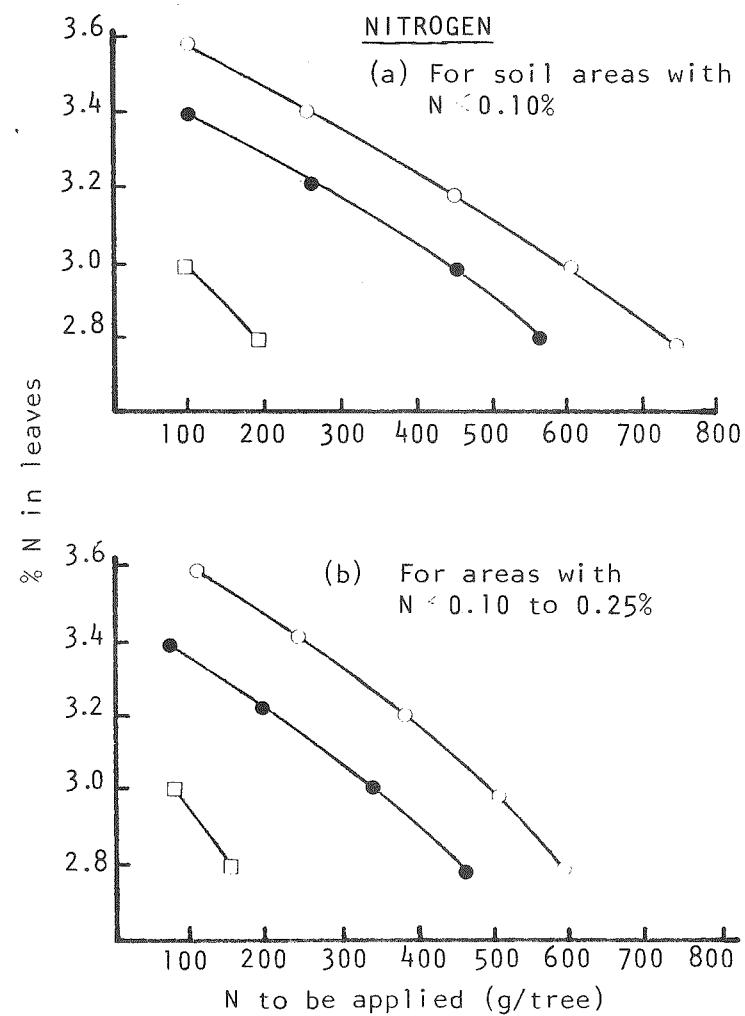


Fig. 1: Rates of application of nitrogen and potassium for mature rubber.

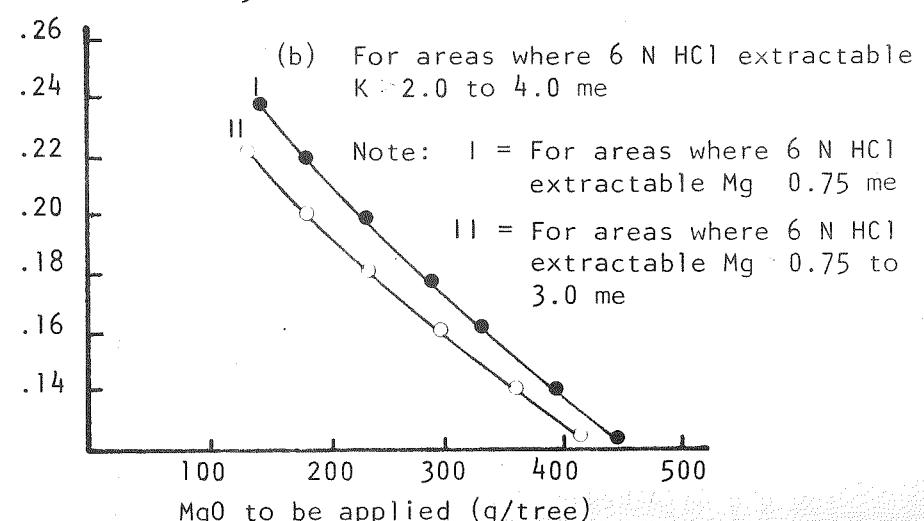
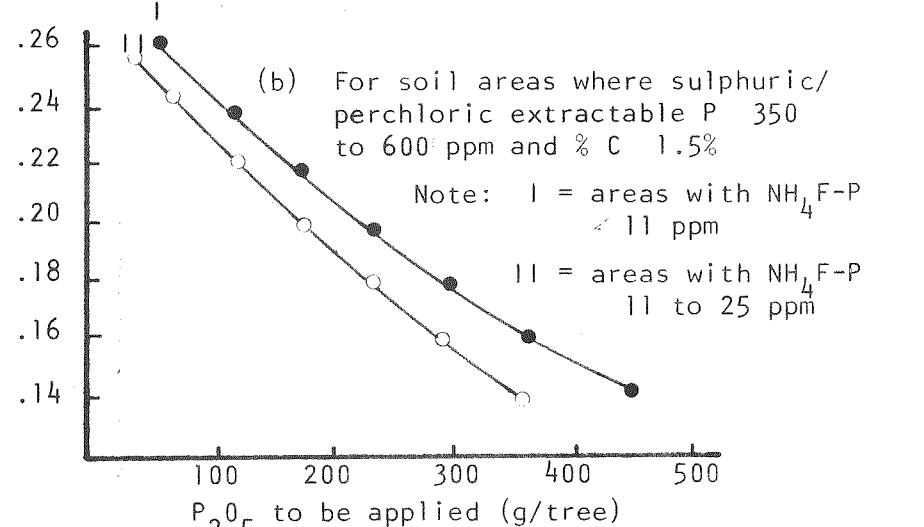
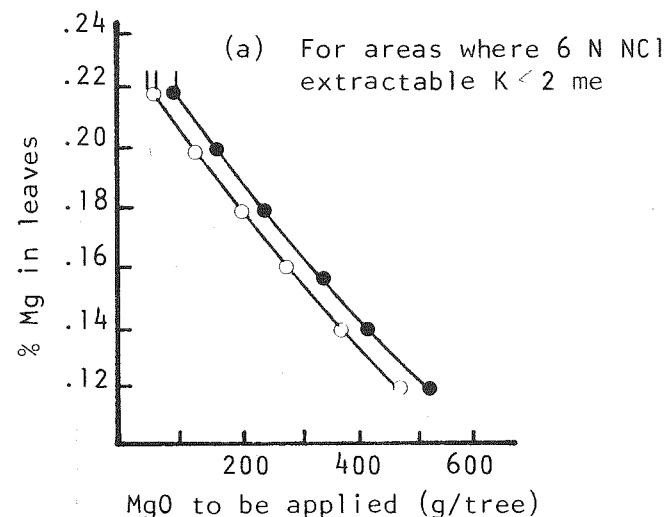
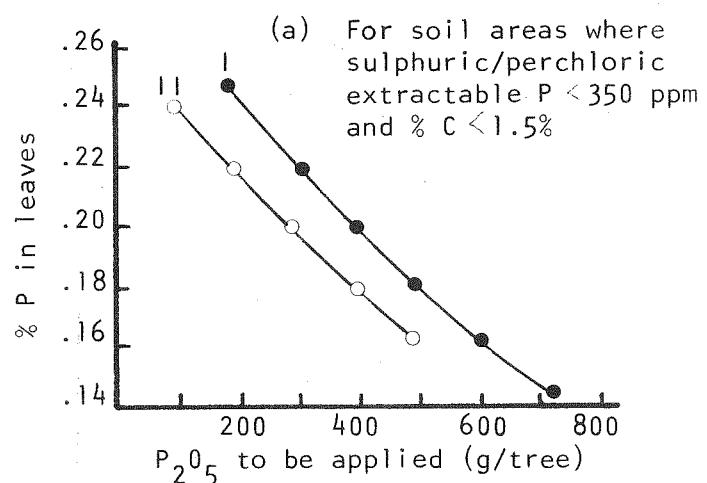


Fig. 2: Rates of application of phosphorus and magnesium for mature rubber.

## INTERPRETATION OF SOIL AND LEAF DATA FOR OIL PALM

by

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## INTRODUCTION

The agronomists have always used leaf analysis data among other factors to draw up manuring programmes. Such other factors are climatic conditions, yields, soils, general field conditions, manurial history and the current agro-management. It has been suggested that foliar analysis per se is not entirely reliable (Green, 1972) and in principle the application of soil and leaf analyses should be advocated. They are in fact two of the many means of investigation into the mineral nutrition of the oil palm. For effective diagnosis, analysis of a host of factors limiting yield performance should be identified.

The purpose of this paper is to outline the need to have an overall view of all the factors influencing yield by proposing a scheme involving total interplay of these factors, and suggesting how this together with the integrated approach using nutrient ratios of leaf data are interpreted for formulating a balance fertilizer programme to improve nutrition of oil palm.

## INTERACTING FACTORS AFFECTING YIELD

Generally, the factors affecting oil palm yield can be grouped as environmental, genetics, soil and agro-management practices. Environmental factors like light, temperature and rainfall should be recorded so that they can be used to calibrate the environment at a given site more accurately. If rainfall distribution

is limiting at a particular period of the year, then we can pinpoint why optimum yield is not sustained.

The amount of rainfall will influence soil moisture. Soil moisture status is important. When the soil is dry during certain periods of the year, we can irrigate and if the soil fertility is poor we can fertilize. If the soil has excessive water, controlled drainage may be practiced.

Whatever we do, the planter has a control over agro-management, e.g. planting the right cultivars, cover establishment, fertilizer application and irrigation. It is most important that all these factors (see Fig. 1) must be recognised during the field visit to the area as they have a direct or indirect effect on plant metabolism and will affect the interpretation of the soil and leaf analyses data.

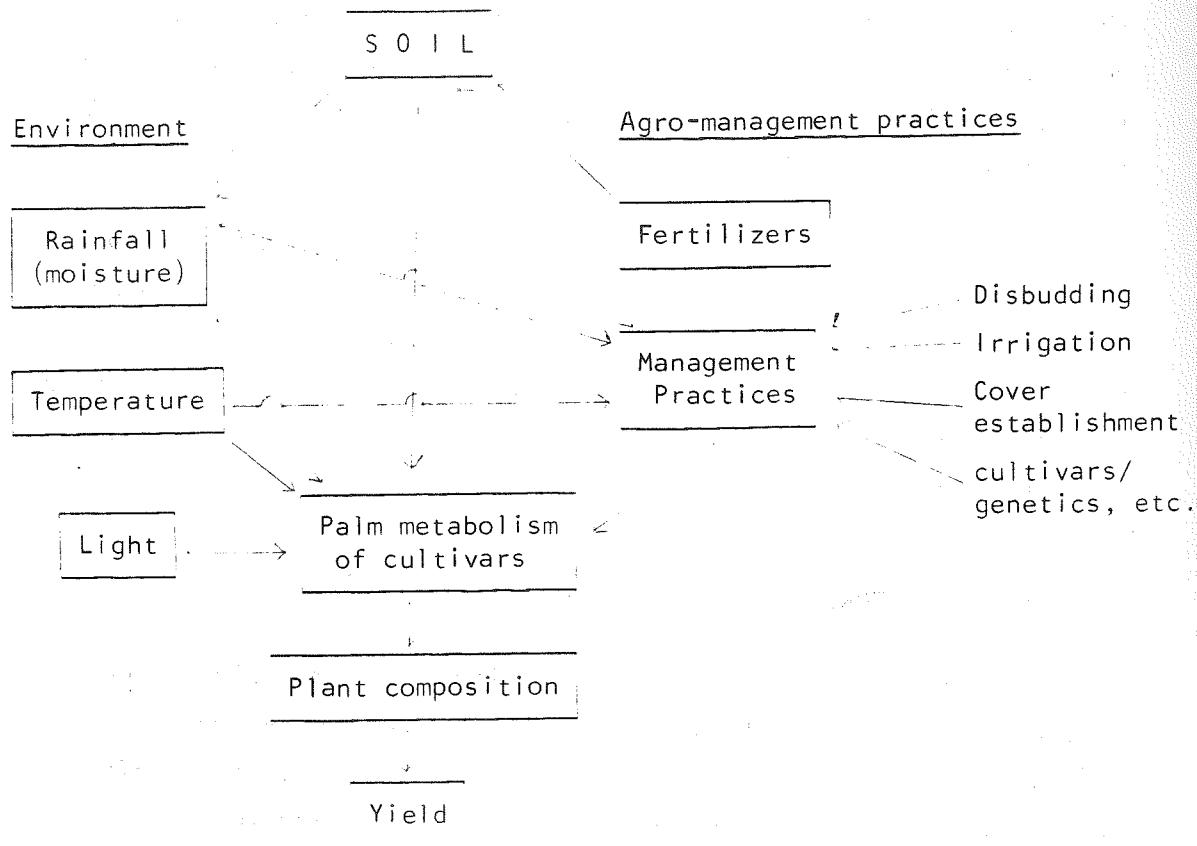


Fig. 1. Interplay of external factors affecting plant composition and yield of oil palm.

## SOME OBSERVED EFFECTS OF ENVIRONMENTAL, SOIL AND AGRO-MANAGEMENT PRACTICES ON LEAF MINERAL STATUS

The following will be drawn upon when interpreting leaf and soil data.

### Environment factors

Considerable information has been accumulated using leaf samples collected from leaf 17. Studies made with variation of leaf levels during short term diurnal changes and long term seasonal fluctuation have been reported. Coulter (1958) and Scheidecker and Prevot (1954) found a significant decrease in leaf N during the day while Rajaratnam (1972) found a steady decline in boron concentration from morning until midday followed by a gradual increase during the afternoon. Our own work (Chemara, 1975) where we did sampling from morning 6.00 a.m. to evening 6.00 p.m., showed that the variations between samples collected at a particular time, were much greater than the variations of leaf levels of samples collected over the day.

In areas where climate is seasonal, leaf K and Ca are higher during the dry season (Broeshart, 1957) and lower during the wet season while leaf N and P behave in the opposite manner. In Malaysia with a less marked dry season, leaf N was found to be 2.54% during a dry period, while increase to 2.74% when the rain returned (Martineau, Knecht and Ramachandran, 1969). Rajaratnam (1972) reported similar changes with boron. Climatic factors also influence leaf nutrient levels indirectly through sex differentiation and abortion rate and during periods of high bunch production a decrease in leaf K and Ca is observed (Coulter, 1958).

### Soil factors

The soil/root system is dynamic (Brewster and Tiner, 1972) and how much nutrient is available in the soil and how rapidly it

can supply water and nutrients to the plant must surely depend on its root system. The oil palm roots are adventitious and can be classified into primary, secondary, tertiary and quaternaries depending on size (Purvis, 1956; Ruer, 1967a, b). Quaternaries are non-signified and are considered together with tertiary roots as the most absorptive roots (Tinker, 1976) though there is little evidence to prove this (Talliez, 1971). Root development elongation and proliferation are strongly affected by manuring (Chan, 1976), and the physical (Tan, 1973) and chemical properties of the soil (Talliez, 1971). Forde (1972) showed that during a dry season a considerable number of fine roots in the surface die off, and by watering during that season, about 3 times the absorption of  $^{32}\text{P}$  was observed in the irrigated plot. The mobility of phosphate is poor in the soil but the results suggest that a considerable proportion of the roots are alive throughout the dry season if watered.

Based on the total nutrient demand of oil palms, described by Ng and Thamboo (1967), and Ng et. al. (1968), a soil must supply 1.3 kg N, 0.2 kg P, 1.8 kg K and 0.3 kg Mg to each palm per year and of this, 60 per cent is permanently removed from the area (Tinker, 1976); whilst in contrast, most soils are able to supply P, 100 to 200 ppm (1.48 kg to 2.96 kg per palm) K, 0.1 to 0.5 me. $100\text{g}^{-1}$  (0.59 kg to 2.96 kg per palm) Mg and 0.1 me. $100\text{g}^{-1}$  (0.18 kg per palm) N annually. Mg will not be met fully by the soil while N will have to come mainly from N fixed by legumes or through its application as fertilizer. For potassium, the demand for replacement is great unless the soil has a strong potassium releasing capacity. Under Malaysian soil conditions Ng (1977) showed that the releasing capacity of the soil has to be taken into account when considering exploiting soil nutrient reserves. Soil K in Malaysian soils may be grouped as labile and non-labile potassium and in general high supply capacity of the marine clays are well demonstrated. Such better potassium supplying power and high buffering capacity is related to the abundance of the clay minerals of the 2:1 lattice model such as montmorillonite and illite. Thus, greater response to potassium fertilization in inland soils of granitic and sandstone origin may be due to the

poor supplying power as these soils have higher 1:1 lattice model clay such as kaolin. Similarly magnesium availability in soil is dependent on the clay mineralogy. Soils with montmorillonitic clays are generally richer and better buffered than kaolinitic soils as indicated by the higher Mg/K exchange ratios (Table 1).

Table 1. Soil types, clay mineralogy and exchangeable Mg/K ratios (Source: Ng, 1977)

Soil type	Clay mineralogy	Exchangeable Mg/K ratios 0 - 8 cm
Rengam	50% kaolin	1.0
Serdang	50% kaolin	1.5
Kuantan	50% kaolin	2.6
Kangkong	50% montmorillonite	15.6
Selangor	50% montmorillonite	12.7

In surface soils, Rengam, Serdang and Munchong have exchangeable Mg/K ratios below 2 which is considered critical by Tinker and Smilde (1963). Thus, a policy of incorporating the dolomitic magnesium limestone as a standard basic dosage for inland soils in Guthrie Estates is a wise recommendation as this will enhance potassium utilisation in the long run.

Most nutrients in soil are affected by leaching. Work done on some Malaysian soils using rainfall simulation technique by Pushparajah, Ng and Ratnasingam (1973) showed there is a considerable scope for reducing fertilizer loss through leaching. Pushparajah et al (1973) suggest that any rainfall exceeding 2 cm per day may cause significant leaching.

For an understanding of the effect of applied fertilizers to the palm, it might be recorded here that Tinker (1976) suggested that potassium, phosphate and magnesium moved to the root by a process of diffusion while nitrogen and calcium are transported by mass flow.

Generally as the soil chemical data from soil samples are not well correlated with oil palm yield (Rajaratnam, Chan and Goh, 1977), perhaps it may be necessary for the soil scientists at this seminar, to look elsewhere, such as the soil solution as a source for nutrient analysis; but this is too variable and often too difficult to be practiced for routine work.

#### Agro-management practices

Fertilizer application has a direct effect on the soil under oil palm. Under African conditions, the fertilizer is applied during the short dry season between two rainy peaks while in Malaysia there is no distinct dry season and application is generally made at any time of the year. Following fertilizer application, Warriar and Piggott (1973) found a rapid rise in leaf N and P concentration in the first 10 weeks after fertilizer application to deficient palms, thereafter leaf level stabilised. Leaf K showed a slow but continuous rise until the next fertilizer application, 6 months later. Rajaratnam (1973) observed that boron in leaf 17 reaches a maximum in 6 to 8 weeks after soil application of borate. Our own trial PF 89 Chemara (1971) showed leaf N was observed to be highest at 13 weeks after application of sulphate of ammonia while that of leaf K at 21 weeks after application of muriate of potash.

Some observed effects of fertilizer on leaf level are shown in Table 2.

#### Cover management

Tropical soils with the exception of peat and recent alluvium have limited soil N reserves. With the use of symbiotic nitrogen fixing bacteria and the cultivation of legumes, leaf N levels and yields have been raised resulted in a saving of 1200 to 1800 kg.ha<sup>-1</sup> of N from legumes over the third to sixth year period of planting (Chemara, 1968; Ng, 1977). The improvement in soil structure

Table 2. The effect of some fertilizer types on leaf levels

Fertilizer	Increase	Decrease	Causes	Source
Ammonium sulphate	N, P	Ca, Mg	Soil competition between $\text{NH}_4^+$ and $\text{Ca}^{++}$ and $\text{Mg}^{++}$	Ochs and Olivin 1976
Urea	N	-	-	Chemara, 1964
Triple phosphate, Rock phosphate	P, Ca	K	K/Ca antagonism in plant	Martin, 1972
Potassium chloride	K	Mg, Ca	K/Mg antagonism in plant	Martin, 1972
Magnesium	Mg	K	Mg/K exchangeable ratio in soil	Smilde and Tinker, 1963
Bunch ash	K, N, P	-	Increased soil pH	Hew & Tan, 1971a

is an additional benefit not quantified. Undesirable weeds like Imperata cylindrica and Mikania cordata have depressed yield by reducing leaf N and P of palms (Gray and Hew, 1968; Highland, 1971). Frequent mowing of interrow reduced leaf N and P (Hew and Tam, 1971). Leaf N is also depressed by presence of other natural covers. Maintenance of bare soil reduces N and K but in areas of high water deficit bare soil gives better water availability and increased leaching of K.

#### Disbudding

In disbudding as carried out in Dahomey, leaf K level improved during the period of disbudding and this is associated with increased root development (Benard and Daniel, 1971). When the disbudded palms come into bearing, leaf K is reduced. In better soil such effect is not observed (Taillez and Olivin, 1971).

## Irrigation

Irrigation improves palm mineral nutrition where nitrogen status in palms remains satisfactory without showing a drop (Desmarest, 1967).

## Cultivars

Ruer (1967b) had showed that Deli Dura palms had a smaller weight of root than Tenera palms of the same age and due to genotype difference nutrients uptake would be different. Tan and Rajaratnam (1977) showed that within Tenera progenies there is indeed genetic variability in leaf nutrient concentration in oil palm. Such variations in leaf N, P, K and Mg should be taken into account in the interpretation of leaf analysis data.

### Effect of age and leaf level gradient according to rank of leaf

Chapman and Gray (1949) were the first to advocate the use of leaf 17 for diagnosis and they showed that P and K of leaf 17 (expressed as percentage of ash) gave better correlation with yield. Coulter (1958) pointed out that this method of expression gave increases in variation not associated with yield. Subsequently the leaf nutrient values were expressed as percentage of dry matter for the major elements N, P, K, Mg and Ca and ppm for the trace elements B, Mn, Mo, Cu, Zn and Fe. Smilde and Chapas (1963) based their selection of leaf 17 on the nutrient levels showing the least variation from palm to palm; and the standard errors for N, P, K and Ca were smallest when compared with those in leaf 1 and 25. In experiments where positive yield responses have been obtained, Rajaratnam, Chan and Goh (1976) demonstrated that leaf 17 can give a satisfactory guide to yield performance.

Leaf nutrient level of individual elements varies with the rank of the leaf, e.g. leaf N increases from leaf 1 to 9 then decreases, the P and K levels decrease whilst Ca and Mg increase. This

necessitates the sampling of a leaf of known rank and the usual choice is leaf 17 as discussed earlier. For immobile element like B, Rajaratnam (1973) suggested the sampling of a number of different ages to determine the nutrient gradient.

#### EXPERIMENTATION

Mineral analysis of plant enables nutrient levels to be defined and compared but does not allow any quantitative judgement. To find out whether a given level is sufficient or not sufficient an experiment has to be done. In the early fifties the type of experiments in Chemara was of  $2^n$  design where n is the number of fertilizer types at 2 levels. By the sixties we conducted experiments with  $3^4$  factorial designs where N, P, K and Mg at 3 levels were tested. We found that for oil palm, N and K are the most important elements giving some 20 to 40% increase in yield while P gave about 5 to 10%. Magnesium did not give any response (Chan and Rajaratnam, 1976). In the seventies and eighties we are now trying the  $4^2$  and  $5^2$  trials where N and K are tested at 4 and 5 levels with P and Mg applied as basal dressings. In our  $3^4$  factorial we also found that leaf N and K plateau off while yield kept on increasing (Lo et. al, 1973). This supports the observed effect that vegetative growth such as leaf, has a higher priority over yield. Based on this, Corley and Mok (1972) suggested that for oil palm the rate of vegetative dry matter production might be a reliable indicator of whether nutrient supply is a limiting factor, since vegetative growth is less affected by short term fluctuation.

#### CRITICAL LEVEL CONCEPT

Based on the results of these experiments various workers have proposed the critical level concept which is defined as the level (expressed as a percentage of dry matter) of an element in a leaf below which the element has every chance of decreasing yield. Some critical levels used for standard leaf analysis of mature palms in different localities and soil types are shown in Table 3.

Table 3. Critical levels for mature palms used by different workers

Source	% Dry Matter				
	N	P	K	Ca	Mg
Ochs & Olivin (1976)	2.50	0.15	1.00	0.60	0.24
Phang (1975)	2.70	0.17	1.10	0.60	0.26
Ng (1969)	2.60-2.70	0.16-0.17	1.10-1.20	0.50-0.60	0.28-0.30

## METHOD OF INTERPRETATION OF SOIL AND LEAF DATA

Interpretation of soil and leaf data has still very much a subjective approach and there is a need to quantify this. An attempt is made here of the leaf data using the integrated approach.

## Soil data

The review of literature shows that there is a lack of knowledge of soil critical ratios on oil palm yield, so far only the exchangeable Mg/K as suggested by Smilde and Chapas (1963) has been applied and the ratio should not fall below 2 for oil palm.

Most of the workers have been looking at the chemical aspects and there is a lack of consideration of the physical aspect. For example, the Segamat series soil is classified as a chemically rich soil but our experience shows that it has poor moisture retention. The long term effect of legume cover on soil in improving soil aggregation in Rengam series soil (Chan *et al*, 1978) need to be extended into other soil types where oil palm is grown. The current work by Universiti Pertanian, Department of Agriculture and Dunlop Research Centre on nutrient removed in run off and water conservation (Mainstone and Tan, per comm., 1978) those of frond arrangement to conserve water (Yeow, per comm., 1978) and our own mulching on Malacca series soil

with empty bunches deserves greater emphasis of research. For it is the poor water retention that is going to limit the oil palm yield production even though nutrients may be sufficient. Until more data on the effects of soil physical properties on root development (Tan, 1973), growth and the long term yield fluctuation are available, it is difficult to develop a system of interpretation of soil chemical data.

#### Leaf data

There is a more definite approach to the interpretation of leaf analysis data. The use of leaf analytical data in the formation of a sound fertilizer programme will require not only the knowledge of the palm nutritional status as reflected by the analytical data, but also, of equal importance are the environmental and agro-management practices as stated earlier. In a nutshell, interpretation relies heavily on the relationship of cause and effect between treatment and visible response of the palm. When a set of leaf analysis data, viewed also over the 3 to 4 proceeding years, is showing a nutrient level below the critical limit, an appropriate corrective dosage of the required fertilizer is recommended to raise the leaf nutrient level close to sustaining optimum yield. A field visit is essential to correlate the leaf analytical data with palm appearance and other relevant observations. This subjective approach is now quantified further.

#### Intergrated nutrient balance approach

The diagnosis using the critical level approach can be difficult when two or more nutrients are deficient. Under such a condition, it is not possible to establish what is more limiting in terms of yield. To overcome this, a system first proposed for rubber by Beaufils (1965) and applied successfully for corn by Sumner (1977) is adapted here for oil palm. The system is simple and is based on the balanced nutrient ratio. It can be applied for any number of nutrients.

To illustrate the application of the intergrated balance concept, 3 nutrients N, P and K are used. Here the indices for N, P and K are first calculated as follows:

$$N \text{ index} = + \left( \frac{f(N/P) + f(N/K)}{2} \right)$$

$$P \text{ index} = - \left( \frac{f(N/P) + f(K/P)}{2} \right)$$

$$K \text{ index} = + \left( \frac{f(K/P) - f(N/K)}{2} \right)$$

$$\text{where } f(N/P) = 100 \left( \frac{N/P}{n/P} - 1 \right) \frac{10}{CV} \text{ where } N/P > n/p$$

$$\text{or } f(N/P) = 100 \left( 1 - \frac{n/p}{N/P} \right) \frac{10}{CV} \text{ where } N/P < n/p$$

in which  $N/P$  = actual value of the ratio in the leaf under consideration

$n/p$  = the mean value of the ratio for the population of high yielding palms in experiment

$CV$  = coefficient of variation for the population of high yielding palms.

The other terms  $f(N/K)$  and  $f(K/P)$  are derived similarly. The mean values of the ratio ( $n/p$ ) and coefficients of variation (Table 4) have been estimated for the population of good yield in 12-year old palms in a  $3^4$  factorial experiments.

These indices have positive and negative values which always sum up to zero as they measure the relative balance among these 3 elements.

The more imbalanced the leaf nutrients, the greater will be the difference between the positive and negative index. But it is the nutrient with a large negative index that will require more fertilizer application to achieve balance with other nutrients. The amount of fertilizer corresponding to the magnitude of the negative

index is currently being examined to see whether a ready reckoner can be produced (Chemara in preparation).

Table 4. Mean diagnostic ratios and their coefficient of variation for population of good yielding 12 years old tenera palms of a  $3^4$  factorial experiment

Ratio	Mean value	CV%
n/p	16.17	4.98
n/k	2.26	8.51
k/p	7.19	7.55

An application of the method will now be illustrated with some examples. In Table 5, the critical levels  $N = 2.70\%$  and  $K = 1.10\%$  will be used.

Table 5. Comparison of critical value approach and integrated balance system of leaf composition in 12 years old Tenera materials

Example	Leaf composition			Integrated indices			Yield (ton.ac <sup>-1</sup> , yr <sup>-1</sup> )
	N%	P%	K%	N	P	K	
1	2.57	.159	.606	51	59	-110	6.26
2	2.59	.172	.809	17	42	-59	7.76
3	2.78	.180	1.218	-4	9	-5	9.02
4	2.97	.175	1.355	3	-10	7	10.02

In Example 1, all 3 nutrients are below the critical level and increases in application of all 3 levels are required. With the integrated approach the large negative K index indicates K is most limiting and imbalance is caused by insufficient K; yield of 6.26 ton.ac<sup>-1</sup>.yr<sup>-1</sup> is low.

In Example 2 by the critical level approach, both N and K are required but by integrated approach only K is most required. There is less imbalance (-59 against -110) and yield of 7.76 ton.ac<sup>-1</sup>.yr<sup>-1</sup> higher.

In Example 3, all levels are again above critical level and no increase is required by critical approach whereas the integrated system shows that relatively to the N and K there is a possibility of P being limiting.

Perhaps an addition of P will raise the yield higher than existing yield of 10 ton.ac<sup>-1</sup>.yr<sup>-1</sup>. In the critical level approach we do not even bother to apply P. Thus, the application of the integrated system will help the agronomist to make better decision of which nutrient to apply and remove any subjectiveness in his recommendation. The system should deserve further development by all who are interested in the interpretation of soil and leaf analyses in oil palm.

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## PROBLEMS ASSOCIATED WITH LEAF AND SOIL ANALYSES TECHNIQUES IN RELATION TO COCOA

by

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### INTRODUCTION

Leaf and soil analyses as diagnostic methods and advisory aids in crop nutrition are well known and are widely used in agricultural system. Techniques have been developed for specific crops and considerable success has been achieved for the main plantation crops in Malaysia such as oil palm, rubber and coconuts. The exception is cocoa.

The problems associated with leaf and soil analyses techniques for cocoa are numerous and form the objectives of this paper. The more salient factors that have significant influence on analysis will be briefly discussed.

### LEAF ANALYSIS AND COCOA NUTRITION

Leaf analysis was introduced to cocoa in 1933 by McDonald (1934) and since then many research workers have tried to use it as a diagnostic aid in assessing the nutrition of cocoa. Almost all workers recognise the problems encountered in the sampling of cocoa leaves and the more salient factors that have significant influence on the mineral composition of the leaf are:

#### Internal factors

- (a) Leaf age
- (b) Leaf position

- (c) Development of new leaves (flushing) and competition effects
- (d) Physiological processes such as flowering and fruiting cycles.

#### External factors

- (a) Fertilizer application
- (b) Light intensity (shade conditions) and its interactions with fertilizers
- (c) Seasonal effects due to climatic conditions
- (d) Soil type.

To elaborate on the problem associated with these factors, findings are quoted from literature and actual experiences in a Dunlop estate in North Johore.

#### Internal factors influencing the mineral composition of the cocoa leaf

##### (a) Effect of leaf age

The age of a cocoa leaf cannot be determined from its position on twigs or branches or by its colour or hardness. This is complicated by the fact that not all branches or individual trees flush at the same time. Leaf age affects the leaf nutrient levels significantly (Table 1).

Although the dry matter content can be used as an index of leaf age (Wessel, 1971), the problem still remains in the sampling of cocoa leaves of the same age at different sampling times.

##### (b) Effect of leaf position

Leaf age and leaf position on a twig or branch are closely related. Preliminary findings from mature monocrop cocoa in a Dunlop estate show significant changes in leaf nutrient levels (Table 2). Similar results have been reported by other research workers (Wessel, 1971).

Table 1. Changes of mineral composition and dry matter content of cocoa leaves with leaf age (After Humphries, 1940)

Age (days) after bud opening	% Dry matter	% of dm					
		Ash	N	P	K	Ca	Mg
14	23.6	7.72	3.70	0.51	2.46	0.46	0.38
42	33.8	7.13	2.31	0.23	1.85	0.84	0.37
70	39.4	9.20	2.38	0.17	1.54	1.23	0.43
98	42.9	11.40	2.32	0.14	1.17	2.09	0.48

Trend with respect to leaf age	I	I	D	D	D	I	I
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I - increasing trend with leaf age.

D - decreasing trend with leaf age.

Table 2. Dunlop experience: Changes in nutrient composition of cocoa leaves with respect to leaf position (After Thong, 1978)

Position of leaf from apex of branch	Leaf analysis data						
	% of dm					ppm of dm	
	N	P	K	Ca	Mg	Mn	Zn
1	2.42	0.18	2.22	0.48	0.39	127	33
2	2.53	0.18	2.26	0.51	0.40	139	29
4	2.43	0.17	2.41	0.53	0.39	152	32
6	2.48	0.15	2.18	0.62	0.41	215	31
8	2.16	0.12	1.56	0.90	0.45	333	24

The current practice in Malaysia is to sample leaf number 4 on the most recently matured flush while leaf number 3 is used in Papua New Guinea (Fahmy, 1977). Thus, there is still some controversy

as to the most satisfactory leaf to sample for meaningful and reproducible results.

(c) Effect of flushing

Work in Brazil has shown that the effect of developing new leaves (flushing) on leaf nutrient composition is significant (Table 3). New flushes exert a competition effect for nutrients.

The lower levels in leaves when the tree is flushing are attributed to migration phenomena of N, P and K from the older to the younger leaves (Humphries, 1940; Wessel, 1970). Since flushing is subject to moisture stress and temperature, irregular flushing cycles can pose serious problems in selection of the proper leaf for chemical analysis.

(d) Effect of flowering and fruiting processes

Limited information is available on the effect of flowering and cropping of leaf nutrient levels. Burridge, et. al (1964) has shown that N, P and K behaved similarly in that their levels tended to be highest each year over the period December to May and lowest over the period July to October. Ca levels varied inversely to those of N, P and K. Mg did not vary greatly from season to season. Figure 1 shows that leaf nutrient levels are affected by flowering and cropping. N, P and K gradually decreased during the cropping season and reached a minimum at the peak of main crop production. It seems that these elements were withdrawn from the leaves to supply developing pods - a competition effect. Thus, for meaningful results from leaf analysis, a specific time must be chosen to sample the leaves. It would seem most logical to sample the leaves at the peak of main cropping season when leaf nutrient levels are at their minimum and when it is most "sensitive" to detect differences.

Table 3. Changes in nutrient composition of leaves with respect to flushing (After Santana and Igue in "Revista Theobroma")

Nutrient element (% of dm)	Physiologically mature leaf: Position in branch	State of flushing	
		without flushing	with flushing
N	1	2.17	1.85
	2	2.18	1.85
	3	2.14	1.71
	4	2.01	1.70
		LSD 5% = 0.19	
P	1	0.18	0.15
	2	0.19	0.15
	3	0.18	0.14
	4	0.17	0.12
		LSD 5% = 0.04	
K	1	1.45	1.06
	2	1.49	1.14
	3	1.41	0.97
	4	1.36	0.89
		LSD 5% = 0.19	
Ca	1	1.66	2.42
	2	1.75	2.47
	3	1.89	2.77
	4	2.00	2.75
		LSD 5% = 0.33	
Mg	1	0.88	0.92
	2	0.91	0.84
	3	0.77	0.88
	4	0.85	0.90
		LSD 5% = 0.16	

## External factors influencing the mineral composition of the cocoa leaf

## (a) Effect of fertilizer application

Numerous workers have reported rapid responses to fertilizer application by the cocoa tree. Burridge, *et. al.* (1964) studied the effect of fertilizers on leaf nutrient levels and they found that the levels of N, P, Ca and Mg increased while the level of K decreased with added fertilizers (Figure 2). However, work from Brazil (Table 4) did not agree well with the findings of Burridge, *et. al.* (1964). The variation in responses is attributed to an interaction between fertilizers and light (or shade) conditions.

Table 4. Changes in leaf nutrient levels with respect to fertilizer in cocoa leaf number 4 (After Santana and Igue in "Revista Theobroma")

Nutrient element	Leaf analysis data	
	% of dm	
	With Fertilizer	Without Fertilizer
N	2.01	1.89
	LSD 5% = 0.19	
P	0.17	0.19
	LSD 5% = 0.04	
K	1.36	1.11
	LSD 5% = 0.19	
Ca	2.00	2.11
	LSD 5% = 0.33	
Mg	0.85	1.02
	LSD 5% = 0.16	

## (b) Effect of light intensity (shade)

It has been reported that shade resulted in an increase in the level of N, P and K and a decrease in the level of Ca in cocoa leaves. There was no significant effects on Mg levels (Figure 3). Sampling leaves from 2 different parts of the canopy on the same tree gave variations to leaf nutrient levels (Table 5).

Table 5. Changes in leaf nutrient levels with respect to cocoa leaves sampled from 2 canopy positions (After Santana and Igue in "Revista Theobroma")

Nutrient element	Position of leaf in canopy	Leaf analysis data	
		% of dm	
		With Fertilizer	Without Fertilizer
N	E*	2.14	2.25
	S**	2.15	2.16
		LSD 5% = 0.14	
P	E	0.18	0.19
	S	0.18	0.20
		LSD 5% = 0.03	
K	E	1.43	1.11
	S	1.42	1.27
		LSD 5% = 0.14	
Ca	E	1.79	0.86
	S	1.88	2.00
		LSD 5% = 0.24	
Mg	E	0.84	1.01
	S	0.86	1.04
		LSD 5% = 0.12	

\* Upper part of the canopy; leaves exposed

\*\* Lower part of the canopy; leaves shaded.

Light intensity or shade condition is a very important factor in the sampling of cocoa leaves for chemical diagnosis since it can cause reactions and interactions with other factors in the physiology of the tree.

(c) Effect of climatic conditions

Cocoa is a rather sensitive plant to fluctuations in climatic conditions and especially to light and soil moisture stress. They affect the physiological processes of the plant e.g. moisture (or rain) and temperature can trigger the mechanisms of flushing and flowering. Alvim and Alvim (1975) referred to this as the phenomenon of "hydro-periodism". Sale (1968) has established that flushing occurs at certain air temperatures. Seasonal effects, therefore, can pose problems to leaf sampling (Figure 1).

(d) Effect of soil type

There is very limited information on the effect of soil type on leaf nutrient composition. In Malaysia, some work has been carried out to assess cocoa nutrition with respect to soil type in a Dunlop estate. Preliminary results seem to indicate that soil type can have considerable influence on leaf nutrient levels (Table 6).

This indicates that soil types should be considered in planning leaf sampling for diagnostic purposes. The problem can be further manifested by the heterogeneity of soils and terrain within the sampling block.

Field application of foliar analysis in cocoa nutrition

Despite its varied problems and precautions to be considered in the sampling technique, foliar analysis has its usage to cocoa. It has reasonable success in detecting gross nutrient deficiencies and in the comparison of treatment in experiments.

Table 6. Dunlop experience: Soil type and average level of nutrients for all leaves in 4-year-old cocoa (after Thong, 1978)

Soil Series	Leaf analysis data						
	% of dm					ppm of dm	
	N	P	K	Ca	Mg	Mn	Zn
Munchong	1.72	0.10	1.38	1.08	0.48	560	22
Prang	2.18	0.16	1.25	2.15	0.55	687	22
Perak	2.12	0.11	0.80	1.34	0.74	995	54
Bungor	2.05	0.11	1.21	0.97	0.44	766	36
Malacca	1.95	0.09	0.54	0.98	0.60	896	22

One such experience is in the correction of Ca deficiency in a Dunlop estate in 1971 (Mainstone, *et. al.*, 1973).

Table 7. Dunlop experience: Correction of Ca deficiency in 1969 cocoa plantings (After Mainstone, *et. al.*, 1973)

Aspect	Leaf analysis (% of dm.)				
	N	P	K	Ca	Mg
Pre-treatment					
(inter-vienal necrosis)	2.39	0.22	2.33	0.21	0.29
S.E.±	0.04	0.02	0.05	0.10	0.02
Post-treatment					
Healthy leaf No. 4	2.36	0.21	2.13	0.36	0.32
S.E.±	0.12	0.02	0.23	0.11	0.15
Healthy leaf No. 8	2.16	0.13	1.74	0.75	0.69
S.E.±	0.05	0.01	0.11	0.12	0.15

Trace element deficiencies can also be detected by leaf analysis. For example, "strap-leaf" or "sickle-leaf" symptoms in cocoa is found to be due to Zn deficiency (Table 8). From the analysis of healthy and affected leaves, it seems that 20 ppm of Zn on a dry matter basis could be the critical value for monocrop cocoa grown on inland soils. Edwards (1978) proposed a critical level of 24 ppm Zn in Papua New Guinea.

Table 8. Dunlop experience: Zn deficiency in cocoa leaves (After Thong, 1973)

Aspect	Leaf analysis on data ppm Zn of dm
Healthy = very young flush	24
Healthy = young leaf	28
Healthy = mature leaf	23
Young "sickle" leaf	17
Mature "sickle" leaf	14

#### Tentative nutrient levels for cocoa leaf

Accepting the various limitations of foliar analysis applied to cocoa, Murray (1967) and Edwards (1978) have proposed tentative nutrient leaf levels in planning fertilizer usage (Tables 9 & 10 respectively). It is obvious that opinions differ probably due to the fact that different leaves are used and that cocoa is grown under varied conditions of climate, soil, density, light intensity and management. This enhances the difficulty of interpretation of leaf analysis data.

Table 9. Proposed leaf nutrient levels by Murray, 1967

Nutrient (% of dm.)	Aspect		
	Deficient	Low	Normal
N	1.80	1.80 - 2.00	2.00
P	0.13	0.13 - 0.20	0.20
K	1.20	1.20 - 2.00	2.00
Ca	0.30	0.30 - 0.40	0.40
Mg	0.20	0.20 - 0.45	0.45

Table 10. Proposed leaf nutrient levels by Edwards, 1978

Nutrient (% of dm.)	Aspect					Above Normal
	Deficient	Critical	Hidden Hunger	Normal		
N	1.90	2.10	2.30	2.70	3.30	
P	0.11	0.13	0.16	0.25	0.35	
K	1.00	1.20	1.60	2.30	3.00	
Ca	0.40	0.60	0.80	1.40	3.00	
Mg	0.30	0.34	0.40	0.60	1.50	
(ppm of dm)						
Mn	15	21	30	50	100	
Zn	20	24	30	40	70	

## SOIL ANALYSIS AND COCOA NUTRITION

Cocoa performs best on deep, sandy clay loam to clay loam soils with good organic matter content, drainage and structure. Smyth (1966) has indicated general soil parameters that are suitable for cocoa while an attempt to classify the suitability of soils in Malaysia for cocoa has been made (Wong, 1974). However, information on the performance of cocoa on various soil types is very limited.

In any case, it is the intention of this paper to present some preliminary findings obtained from Dunlop's investigations on the growth and nutrient composition of 4-year-old cocoa with respect to 5 inland soils in Malaysia, Tables 11 and 12 (Thong, 1978).

Table 11. Dunlop experience: Soil type and dry matter production by 4-year-old cocoa (After Thong, 1978)

Soil Series	Whole Plant (kg)	Root System (kg)	Root System as % Whole Plant
Munchóng	11.70	2.30	20.3
Prang	10.76	2.56	23.8
Perak	16.77	3.33	19.9
Bungor	10.00	1.88	18.8
Malacca	10.42	1.68	16.1

Results indicate that soil type has considerable influence on the growth and nutrition of cocoa. Fertilizer K and N responses have already been reported for mature cocoa on Bungor Series (Mainstone and Thong, 1978). Thus, it would seem reasonable to assume that soil analysis could assist in estimating fertilizer requirements for cocoa. In Brazil (Alvim, 1975) and Jadin (1975) have used soil analysis as a diagnostic and advisory aid in their cocoa fertilizer programmes.

Table 12. Dunlop experience: Soil type and total nutrient composition in 4-year-old cocoa (After Thong, 1978)

Soil Series	Total nutrient content (g per plant)						
	N	P	K	Ca	Mg	Mn	Zn
Munchong	108	10	161	90	35	2.5	0.3
Prang	128	18	161	143	38	2.5	0.3
Perak	196	16	132	153	87	7.2	1.4
Bungor	98	10	132	54	26	2.8	0.3
Malacca	113	11	114	87	36	3.0	0.3

Tentative nutritional standards for cocoa soils

As with leaf analysis data, there is still no general agreement in the nutritional standards for cocoa soils. This seems to be unavoidable due to the varied conditions under which cocoa is cultivated.

Be it as it may, Hardy (1960) and Edwards (1978) have proposed tentative nutritional standards for cocoa soils (Table 13 & 14 respectively).

Table 13. Provisional standards for cocoa soils (0 - 6" layer) by Hardy (1960)

Nutrient Status	pH	Total N (%)	C/N Ratio	Available (ppm)		Exchangeable Bases (me.100g <sup>-1</sup> )		
				Truog P <sub>2</sub> O <sub>5</sub>	Exch. K <sub>2</sub> O	CaO	MgO	K <sub>2</sub> O
High	7.5	0.35	11.5	120	260	24.0	6.0	0.55
Medium	6.5	0.20	9.5	60	170	12.0	3.0	0.35
Low	5.0	0.05	7.5	20	90	4.0	1.0	0.20

Table 14. Tentative standards for cocoa soils by Edwards (1978)

Nutrient Status	pH	Total N (%)	Available (ppm)	Exchangeable Bases (me.100g)		
				P	K	Ca
Above normal	7.5	0.80	9.0	1.2	13.0	8.0
Normal	6.5	0.60	7.0	0.8	8.0	3.0
Hidden hunger	5.5	0.40	5.5	0.5	5.0	1.0
Critical	5.0	0.30	4.5	0.3	3.0	0.5
Deficient	4.0	0.20	3.5	0.2	1.0	0.2

## COMMENTS

1. Leaf and soil analyses when applied to estimate cocoa nutrient requirements, face numerous problems and limitations in their sampling techniques. This is probably due to the diverse conditions of soil, climate, light, density and management levels under which cocoa is cultivated. Their diagnostic value in cocoa is, thus limited.

2. Despite these limitations, both leaf and soil analyses have useful applications in the detection and correction of gross nutrient deficiencies.

3. However, inspite of the considerable amount of research on cocoa leaf and soil analyses, there are considerable refinements required in these techniques. Thus, to-date there are no satisfactory leaf and soil analyses methods available in the formulation of realistic fertilizer programmes for all conditions. More research is required.

4. An integrated approach involving leaf and soil analyses may be the answer to cocoa nutrition studies.

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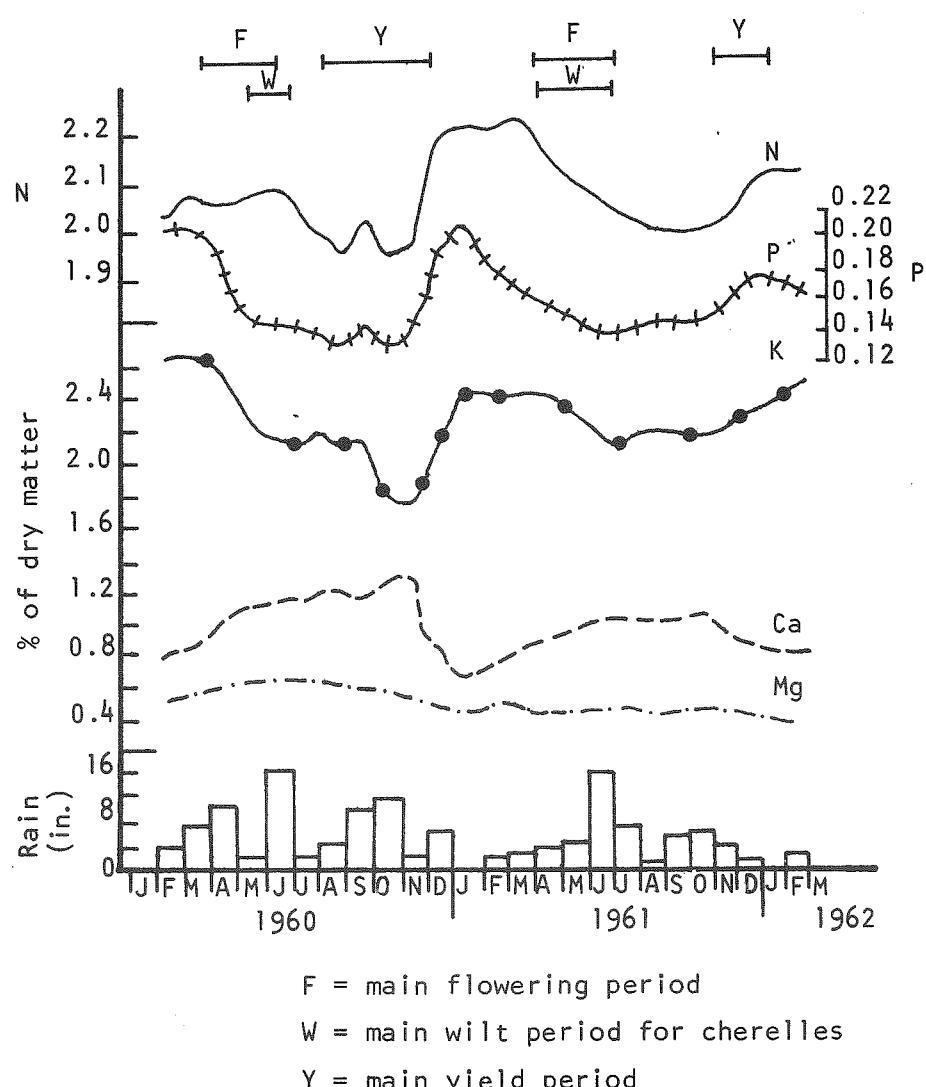


Fig. 1: Leaf contents of N, P, K, Ca and Mg as affected by cropping season and rainfall (After Burridge, et. al, 1964)

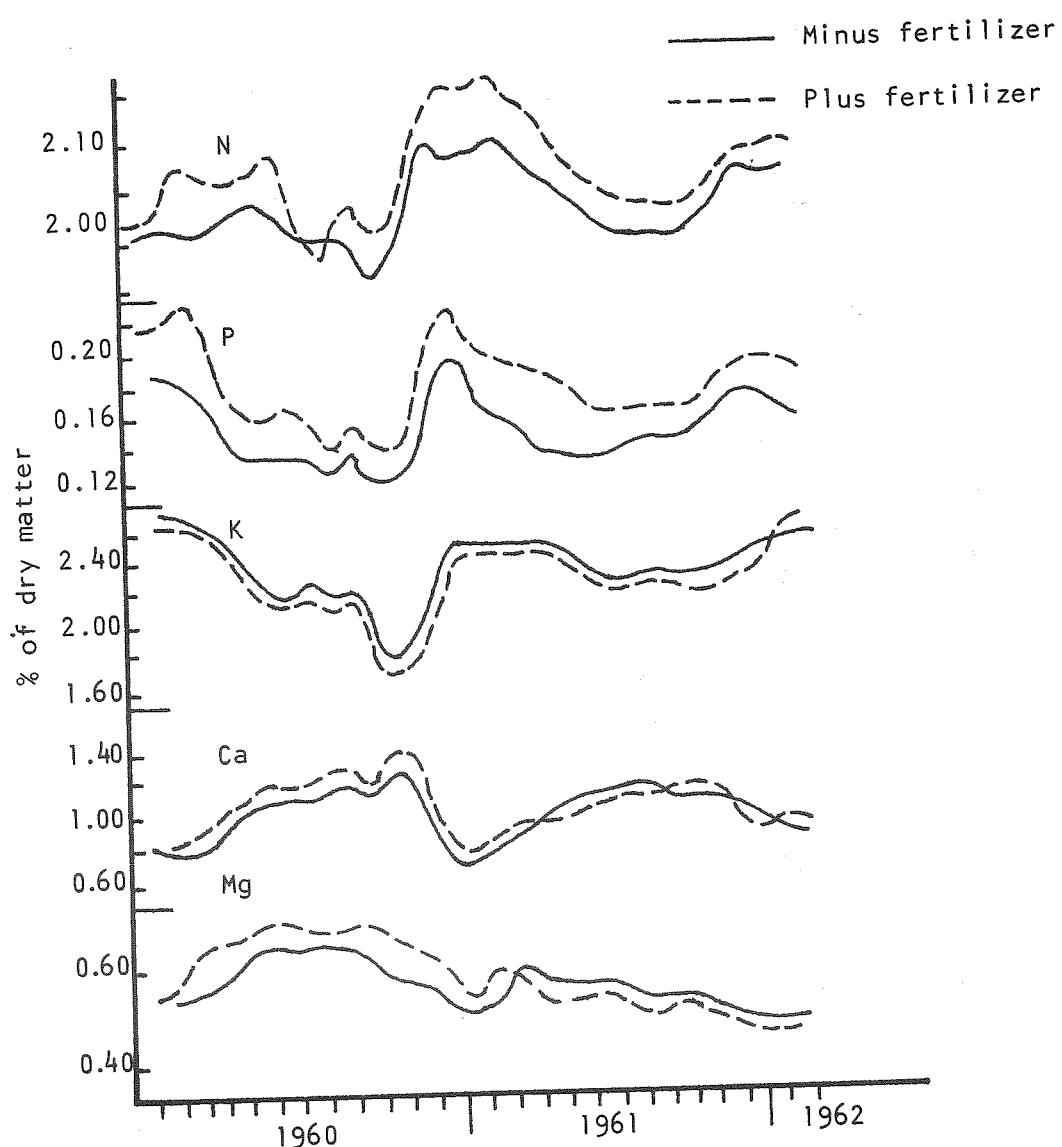


Fig. 2: Effect of fertilizer on leaf nutrient levels in cocoa (After Burridge, *et. al*, 1964)

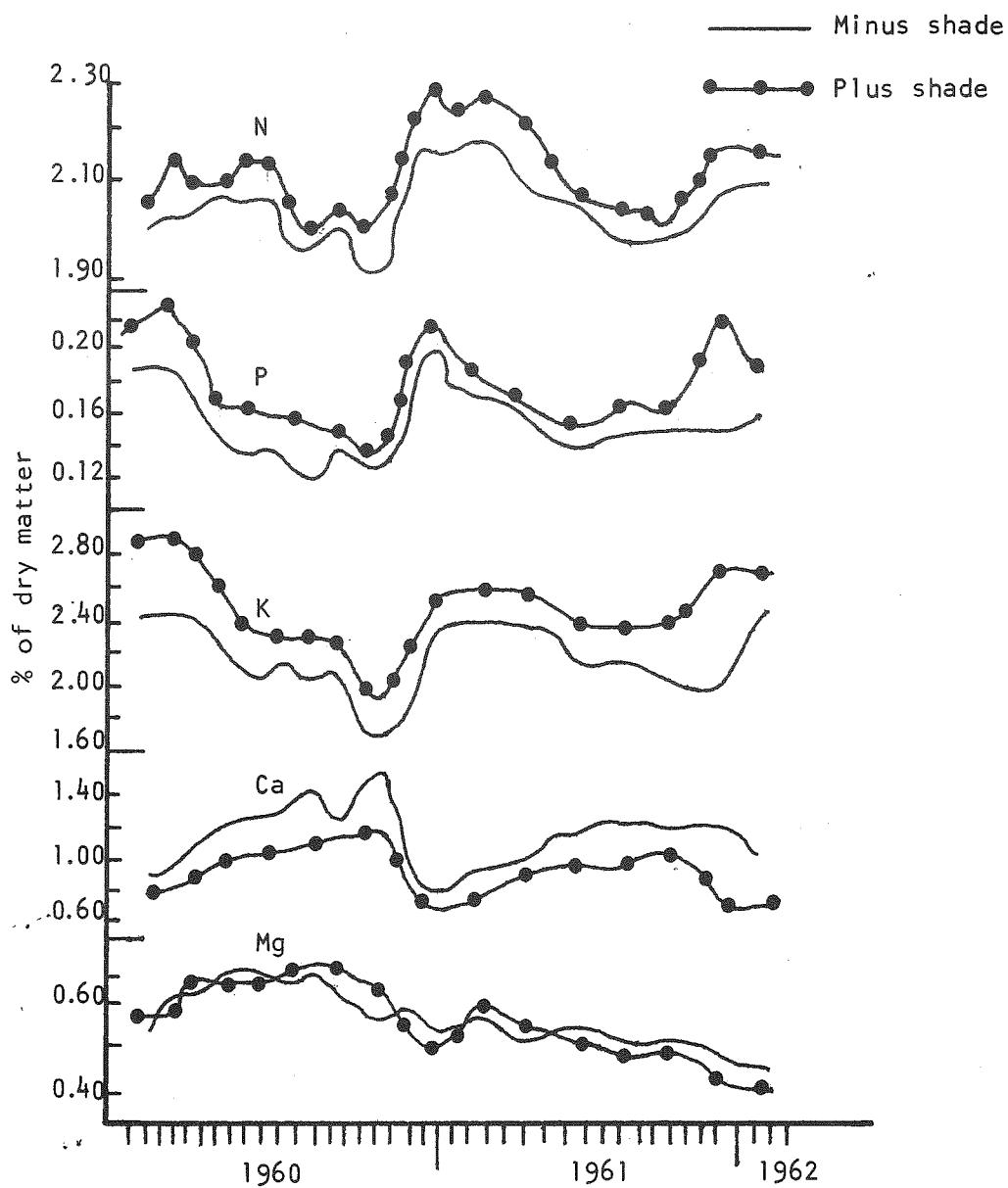


Fig. 3: Effect of shade on leaf nutrient levels in cocoa (After Burridge, *et. al*, 1964)

POTENTIAL USE OF SOIL AND LEAF ANALYSIS DATA FOR  
GROUNDNUTS AND OTHER FIELD CROPS IN MALAYSIA

by

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SUMMARY

A Cereal/Grain Legume Nutrition Project recently started by MARDI to ascertain fertilizer requirements of field crops on different soils in Malaysia is briefly described. Some preliminary results with groundnuts suggest that P and K fertilizer requirements can be predicted from soil buffer curves but not from conventional soil tests.

INTRODUCTION

At present there exists very limited information on the varying fertilizer requirements of field crops on different soils in Malaysia, and to provide more information on this topic a Cereal/Grain Legume Nutrition Project has been recently started by MARDI. In this paper the project is briefly outlined and some initial results from groundnut trials are presented and discussed.

FERTILIZER TRIALS

Approximately 20 fertilizer trials testing three levels of N, P and K fertilizers and lime in factorial combination have been established on a range of soils in areas throughout Peninsular Malaysia where field crops are commonly grown. A yield response function which takes into account main linear and quadratic effects

and linear two and three factor interactions is fitted to the plot yield data, from which the following information is deduced:

- 1) Crop yields without individual fertilizers  
(at optimum levels of other fertilizers).
- 2) Yield responses to individual fertilizers  
(at optimum levels of other fertilizers).

It is intended to continue these trials on the same sites for three years, two crops including either maize, groundnuts, mungbeans or soybeans, being grown each year.

#### SOIL ANALYSIS

The nutrients available to a crop are held in the soil solution and adsorbed on the surface of soil particles, and it is necessary to measure the nutrients in both these phases in order to fully characterise the nutrient status of a soil. The required information is provided by buffer or sorption curves which in this work are determined by procedures similar to those described by Beckett (1964). Fox and Kamprath (1970) and Van Raij and Peech (1972) for K, P and H respectively. Essentially all the methods involve equilibrating the soils with solutions containing concentration increments of the nutrient (or ion) under study. The final concentration in the solution is plotted against the amount of nutrient (or ion) gained or lost by the soil. As a major treatment in the trials was lime, the basic equilibrating solution chosen for K and P was 0.01 M  $\text{CaCl}_2$  (but for H the basic solution was 0.01 M  $\text{NaCl}$ ).

Soil samples (0-20 cm) were collected at the start of each trial and were analysed by the above methods. From the buffer curves obtained two properties were then deduced for each nutrient:

- 1) The equilibrium soil solution concentration (which is the concentration of the solution which undergoes no change when shaken up with the soil).
- 2) The current buffer capacity of the soil (which is the slope of the buffer curve at the equilibrium solution concentration).

The first property indicates the immediate availability of a nutrient. However, the availability over a period of time also depends on the second property which indicates the ability of a soil to maintain the initial soil solution concentration, so that yield would be expected to be positively related to both properties. The second property also indicates the increase in soil solution concentration which would be brought about by addition of a given amount of fertilizer. The higher the buffer capacity the smaller the increase, so yield response would be expected to be inversely related to this second property, and also to the first property if response falls off the higher the yield.

#### LEAF ANALYSIS

For annual crops leaf analysis is of less use than soil analysis, since action based on the results can be taken only for the following crops. In addition interpretation of leaf analysis results is more difficult, since an imbalance of one nutrient alters the levels of other nutrients. From leaf analysis results, one can diagnose safely only the most deficient nutrient - deductions about the availability of the other nutrients cannot be made from leaf nutrient data with confidence until the imbalance of the first nutrient is corrected. Thus leaf analysis gives limited instant information, but used as a supplement to a research programme may give very valuable information over a period of time. In the present project leaf samples are collected from selected treatments of all maize and groundnut trials. In the case of groundnut the sixth

leaf (plus petiole) from the base of the main stem is collected at the start of flowering.

#### INITIAL RESULTS FOR GROUNDNUT TRIALS

During the first half of 1978, five well managed groundnut trials were harvested. Their location and soil type is indicated in Table 1.

#### Potassium

Groundnut yields without K fertilizer and responses to K fertilizer at optimum levels of other fertilizers deduced from the fitted response functions are shown in Table 2 together with soil K data, whilst K buffer curves are depicted in Figure 1. Yields without K fertilizer ( $Y_1$  kg.ha<sup>-1</sup> grain) were poorly related to the conventional exchangeable K soil test ( $r = 0.37$ ) as shown in Figure 2, and to the equilibrium soil solution concentration, but showed an excellent quadratic relationship ( $r = 0.99$ ) with the soil K buffer capacity ( $X_1$  me. 100g<sup>-1</sup>) as shown in Figure 3. The fitted regression equation was:

$$Y_1 = 894.5 - 2105.2X_1 + 9003.3X_1^2$$

Response to 30 kg.ha<sup>-1</sup> K<sub>2</sub>O fertilizer ( $Y_2$  kg.ha<sup>-1</sup> grain) was not well related to any single soil measurement, but the multiple correlation with both the soil K buffer capacity and the equilibrium soil solution K concentration ( $X_2$  ppm) was significant ( $r = 0.92$ ). The fitted regression equation was:

$$Y_2 = 410.6 + 1386.5X_1 - 9.19X_2$$

and the predicted relationship between response and soil solution K concentration at two different buffer capacities is illustrated in Figure 4 where the original data is also shown.

### Phosphorus

Groundnut yields without P fertilizer and responses to phosphate fertilizer are shown in Table 2 along with soil P data, whilst P buffer curves are shown in Figure 5. Due to the lack of previous P fertilization the equilibrium soil solution P concentration was generally too small to measure (<0.005 ppm). Langmuir adsorption equations were fitted to the buffer curve data from which maximum buffer values were calculated.

Soil P extracted by the Bray No. 1 method accounted for more variation in groundnut yield without P fertilizer ( $r = 0.67$ ) than the Olsen method or maximum buffer capacity values, but the correlation was not statistically significant. The spread of data is shown in Figure 6 together with the line of best fit. Response to 50 kg.ha<sup>-1</sup> P<sub>2</sub>O<sub>5</sub> fertilizer ( $Y_3$  kg.ha<sup>-1</sup> grain) was however, found to be highly significantly correlated with soil P maximum buffer capacity ( $X_3$  = ml. g<sup>-1</sup>) especially when a logarithmic relationship was tested ( $r = 0.99$ ) as shown in Figure 7. The fitted regression equation was:

$$Y_3 = 1663 - 266 \log_e X_3$$

### Lime

Groundnut yields without lime and responses to lime are shown in Table 3 together with relevant soil data, and soil pH buffer curves are shown in Figure 8. Yields without lime ( $Y_4$  kg.ha<sup>-1</sup> grain) were most significantly related ( $r = 0.89$ ) to soil pH ( $X_4$ ). The fitted regression was:

$$Y_4 = 768.3X_4 - 2708$$

and is depicted in Figure 9 together with the original data. Response to lime was not found to be well related to any of the soil properties measured.

### Leaf Analyses

Groundnut yield responses to P fertilizer and leaf P results from trials carried out by the senior author in Uganda (Foster, 1979) are shown in Figure 10, from which a critical leaf P level of 0.29% can be deduced. Results from the present trials carried out in Malaysia are also shown in Figure 10, from which it can be seen that although most leaf P levels are below the critical level, few appreciable responses to P fertilizer were obtained. This is because only selected treatment plots were sampled and P responses in these plots were often restricted by an imbalance of other nutrients. However, given sufficient data a critical level can usually be deduced above which fertilizer responses are never obtained, but below which a response may be obtained depending upon the level of other nutrients. Leaf K results obtained in the present trials are shown in Figure 11, from which a very tentative critical level of 2.7% may be deduced.

### DISCUSSION

The ultimate aim of the Cereal/Grain Legume Nutrition Project is to be able to predict the fertilizer requirements of field crops in any particular environment in Malaysia from soil and climatic information. In this paper the soil information considered has been chemical analyses, particularly soil buffer curves and the potential usefulness of these measurements has been examined in the few groundnut trials carried out so far.

To be able to predict the requirement of an individual fertilizer it is necessary to know the slope of the response curve and the current position on the response curve. The results presented in this paper indicate that the slope of P and K response curves can be predicted from soil buffer curves. In particular P responses appear to be very closely related to soil P maximum buffer capacity values. Unexpectedly K response was found to be positively related to K buffer capacity, which perhaps indicates that the ability of a soil to retain added K is particularly important under Malaysian conditions.

The present results also indicate that yield without K fertilizer, which defines the current position on the response curve can be satisfactorily predicted from the soil K buffer capacity but not from conventional soil K tests. A similar finding for Malaysian soils has been reported by Singh and Talibudeen (1969) who found that plant K uptake in pots was best related to the soil K buffer capacities. Yields without K fertilizer and lime were moderately well related to soil P extracted by the Bray No. 1 method and to soil pH respectively, indicating that these conventional tests may be of some value. However, the correlation with the soil P test was not significant, and in soils which have received P fertilizer the equilibrium soil solution P concentration can be expected to be better related to yields than conventional P extraction methods.

#### ACKNOWLEDGEMENT

The assistance of Lembaga Pertubuhan Peladang staff in Kelantan, Trengganu, Pahang and Kedah, and of Syarikat Ubiyu Malaysia Berhad in Perak, who helped manage the trials is gratefully acknowledged. Thanks are also due to many MARDI staff associated with the project, particularly Encik Abdul Rahman Haron, Encik Noor Rawi Abu Bakar, Encik Mohd. Khairol Mohd. Ariff and Encik Mohd. Azmi Abdullah who helped establish and supervise the trials. We also thank Encik B. Gopinathan for soil survey advice and Dr. Heng Chin Kok for analysis of leaf samples.

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Table 1. First season 1978 groundnut fertilizer trials.

Trial No.	State	Area	Soil classification	Soil Series
5	Pahang	Rompin	Aquic Tro-pohumod	Rompin
12	Kedah	Merbok	Tropaquent	Coll.-alluvium
16	Perak	Sitiawan	Tropohemist	Muck
25	Kelantan	Pasir Mas	Oxic Dystopept	Tok Yong
28	Trengganu	Kuala Berang	Fluventic Tropopsamment	Telemong

Table 2. Groundnut yields and responses to K fertilizer and soil and leaf K data for each trial

Trial No.	Yield without K fertilizer	Response to 30 kg.ha <sup>-1</sup> K <sub>2</sub> O				Leaf % K
	Groundnut grain (kg.ha <sup>-1</sup> )		Exch. 'K me.100g <sup>-1</sup>	Equilibrium soln. conc. ppm K	Buffer capacity* me.100 ppm <sup>-1</sup>	In absence of K fertilizer
5	1716	845	0.18	23.5	0.44	3.09
12	814	256	0.09	45.0	0.08	1.70
16	1281	181	0.36	81.5	0.38	2.66
25	747	76	0.18	39.0	0.15	2.56
28	1007	282	0.13	55.0	0.27	2.73

(\* at equilibrium soln. concentration)

Table 3. Groundnut yields and responses to P fertilizer and soil and leaf P data for each trial

Trial No.	Yield without P fertilizer		Response to 50 kg.ha <sup>-1</sup> P <sub>2</sub> O <sub>5</sub>		Soil P data		Leaf % P	
	Groundnut grain (kg.ha <sup>-1</sup> )		Extr. P (Bray No. 1) ppm	Maximum buffer capacity ml.g <sup>-1</sup>	In absence of P fertilizer			
5	1718	444	16.0	105		0.31		
12	1000	577	9.8	58		0.19		
16	1472	-423	21.3	1978		0.23		
25	802	-105	14.8	1050		0.27		
28	804	245	3.6	185		0.23		

Table 4. Groundnut yields and responses to limestone, soil acidity and leaf Ca data for each trial

Trial No.	Yield without lime		Response to 2 t ha <sup>-1</sup> GML	Soil data			Leaf % Ca In absence of limestone
	Groundnut grain	(kg.ha <sup>-1</sup> )		Exch. Ca me.100 g <sup>-1</sup>	pH	pH buffer capacity me/pH unit	
5	1231		487	2.22	4.98	1.05	1.69
12	922		494	0.83	4.48	0.62	0.70
16	337		620	0.39	4.00	4.23	1.03
25	675		-311	1.04	4.70	0.96	0.80
28	1036		162	2.84	4.98	0.86	1.13

(GML = Ground magnesium limestone)

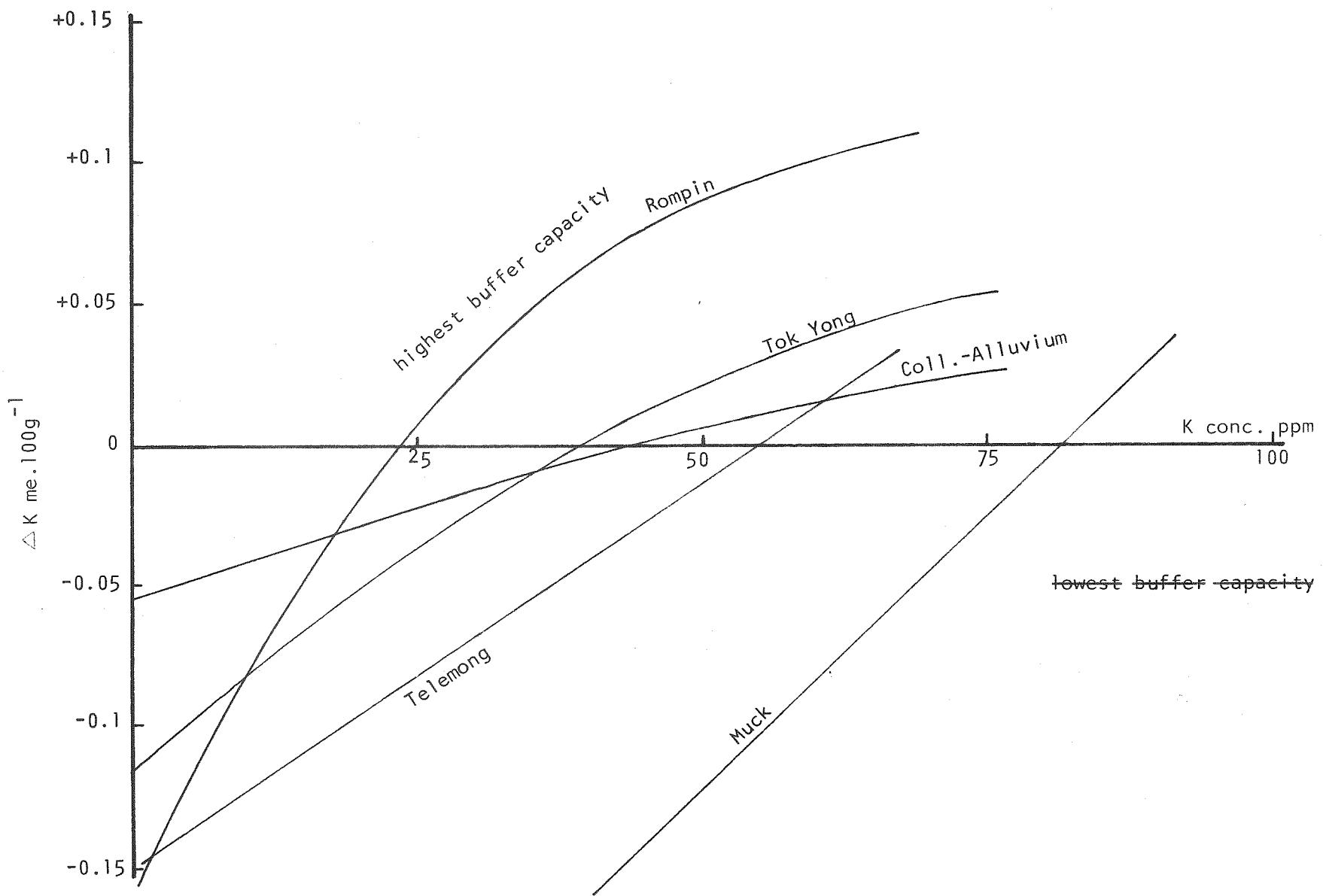


Fig. 1: Soil K buffer curves

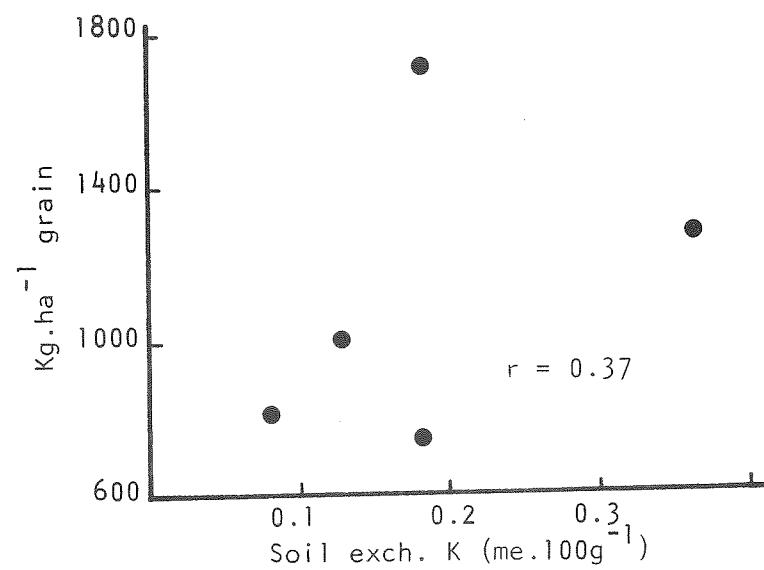


Fig. 2: Groundnut yield without K fertilizer in relation to soil exch. K

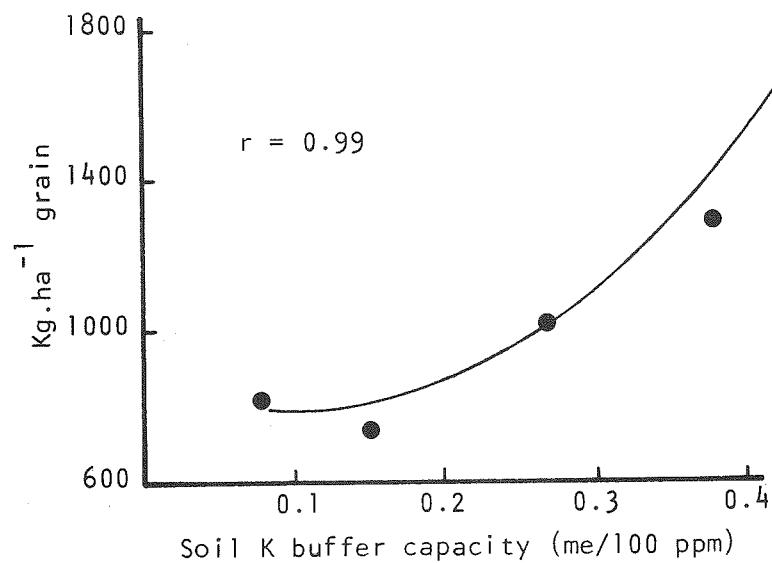


Fig. 3: Groundnut yield without K fertilizer in relation to soil K buffer capacity

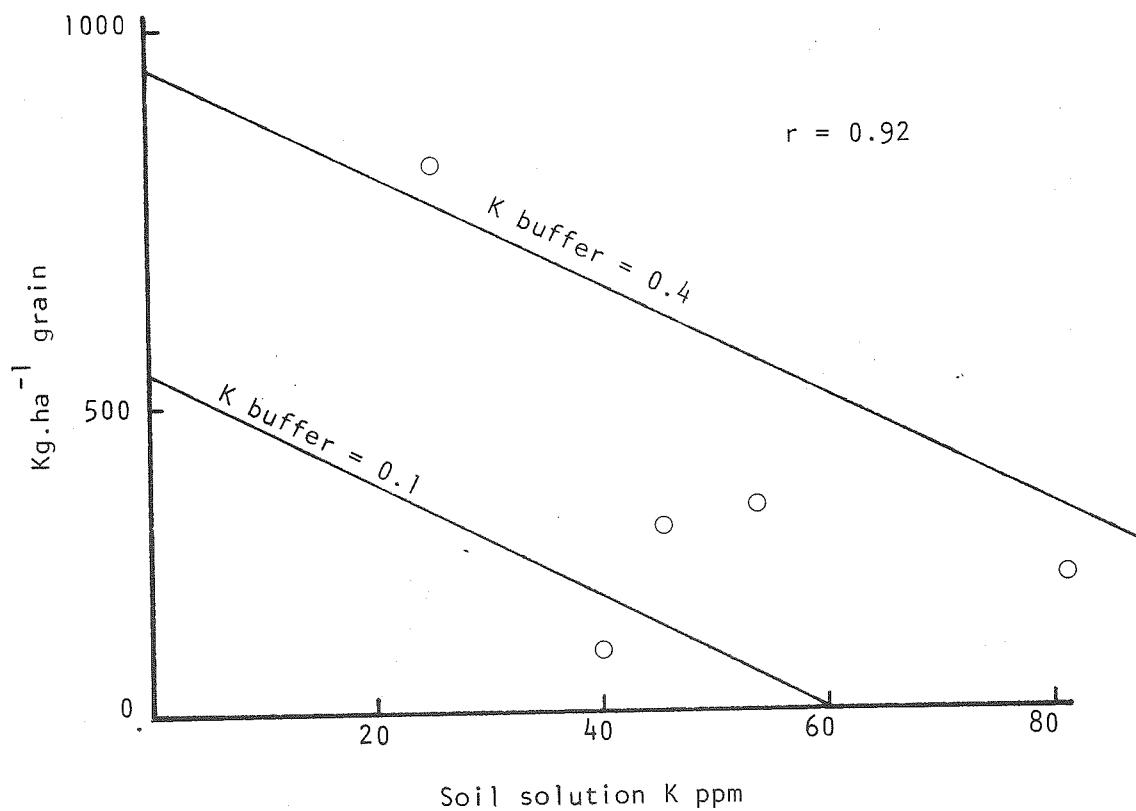


Fig. 4: Groundnut yield response to  $30 \text{ Kg.ha}^{-1} \text{K}_2\text{O}$  in relation to equilibrium soil solution K concentration.

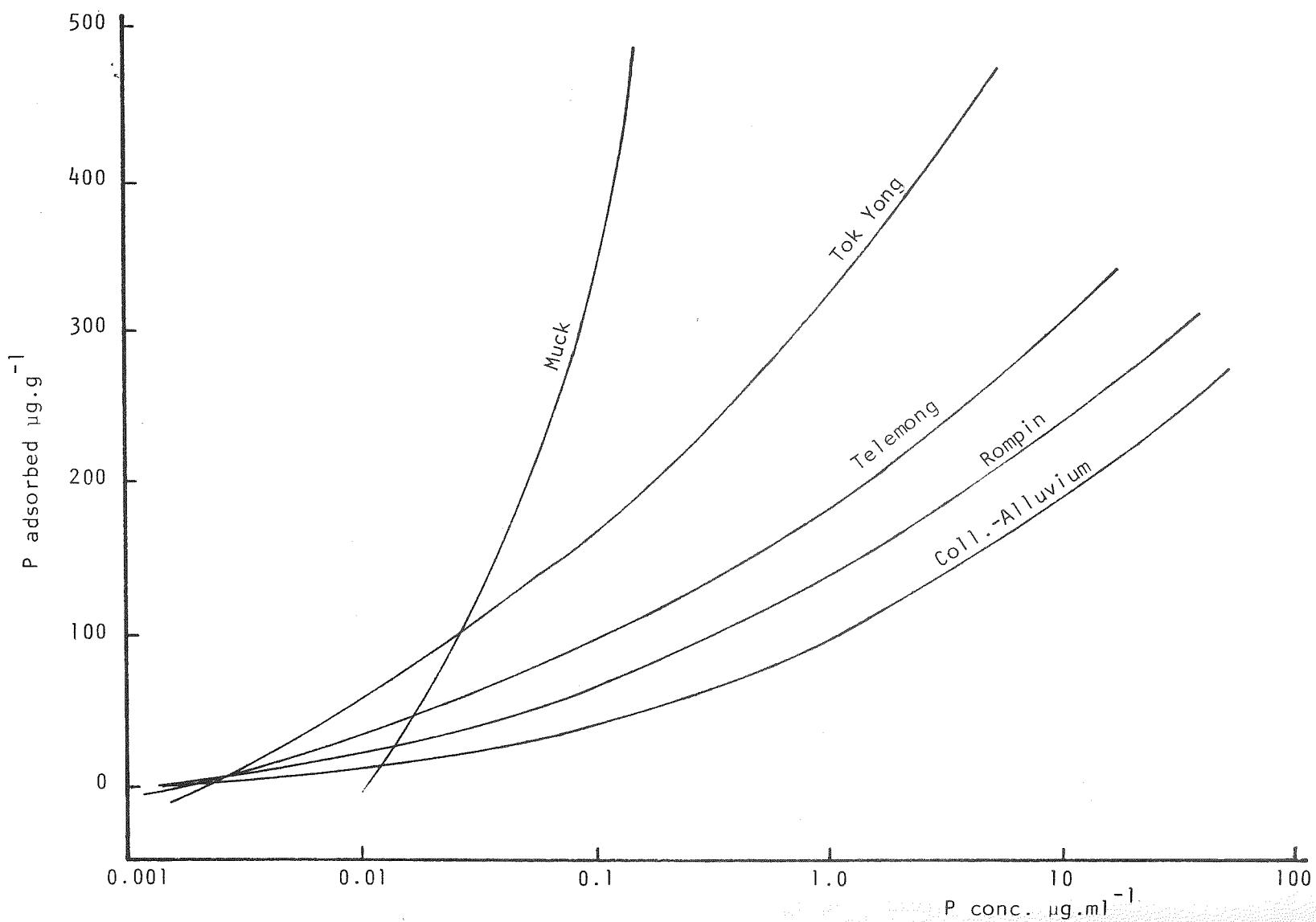


Fig. 5: Soil P buffer curves

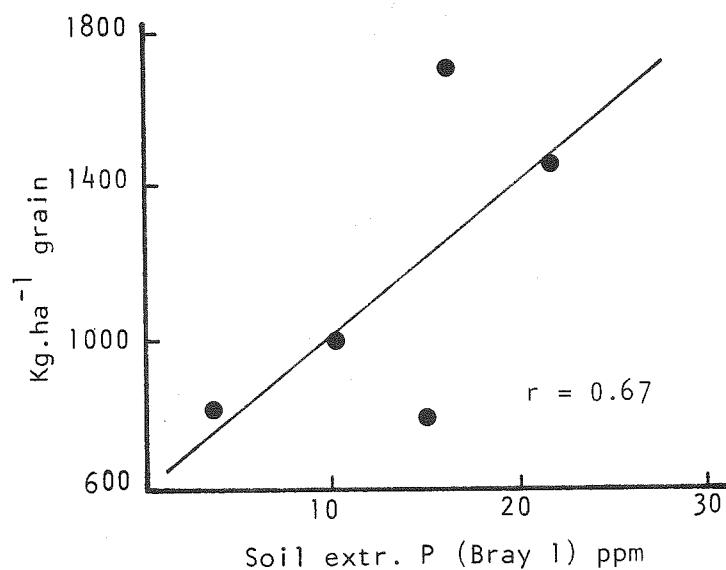


Fig. 6: Groundnut yield without P fertilizer in relation to soil extr. P (Bray No. 1)

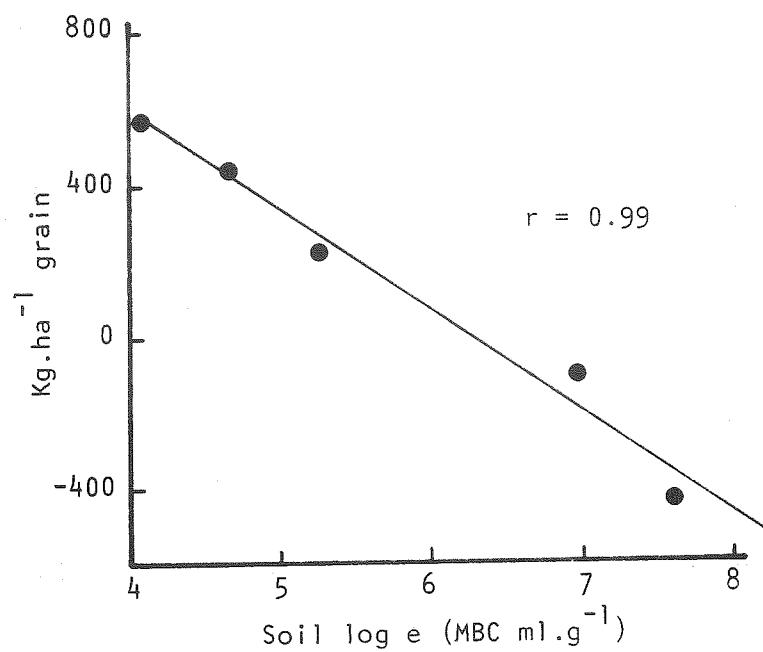


Fig. 7: Groundnut yield response to 50 kg.ha<sup>-1</sup> P<sub>2</sub>O<sub>5</sub> in relation to soil P Maximum Buffer Capacity (MBC)

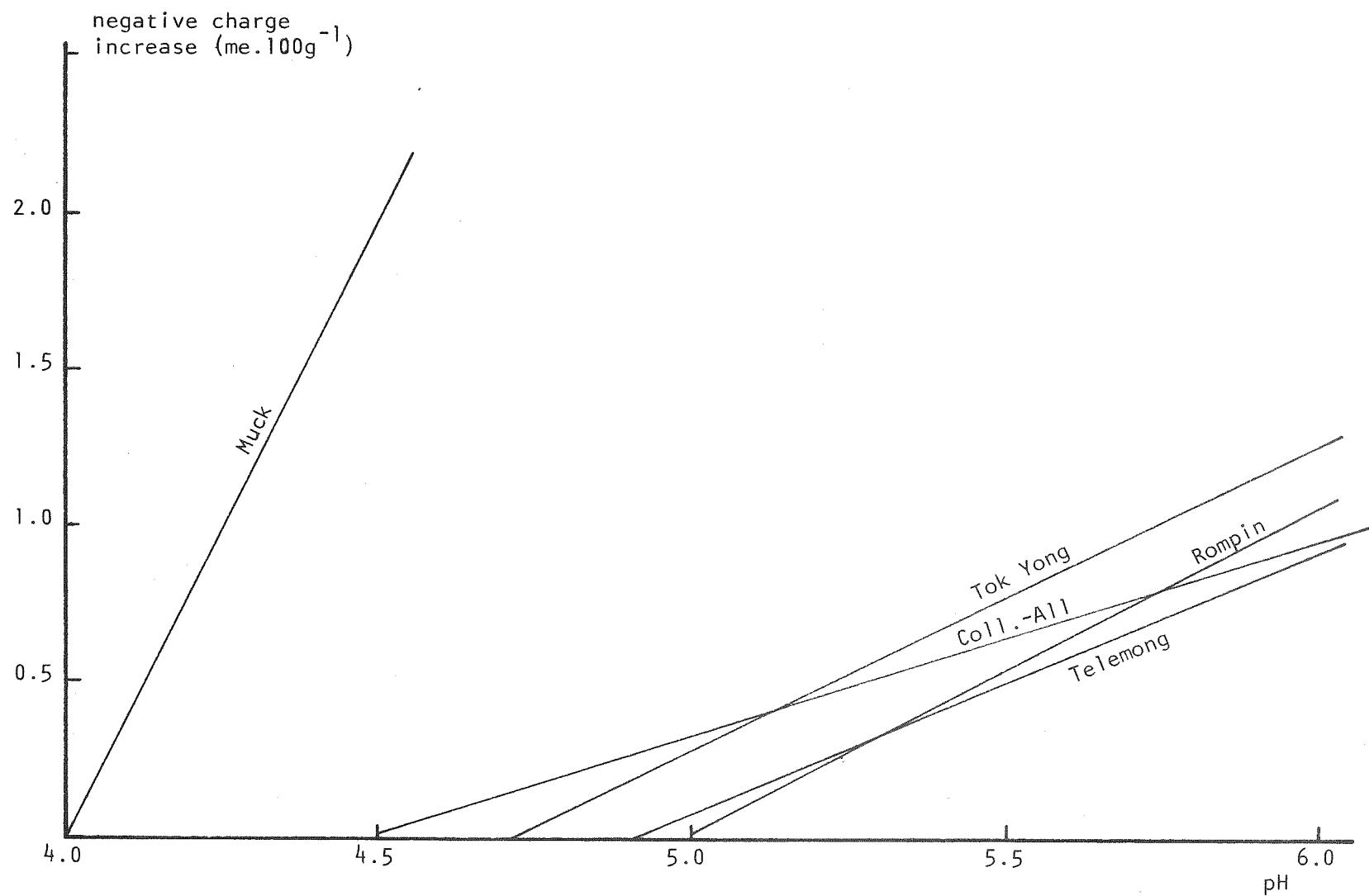


Fig. 8: Soil pH buffer curves

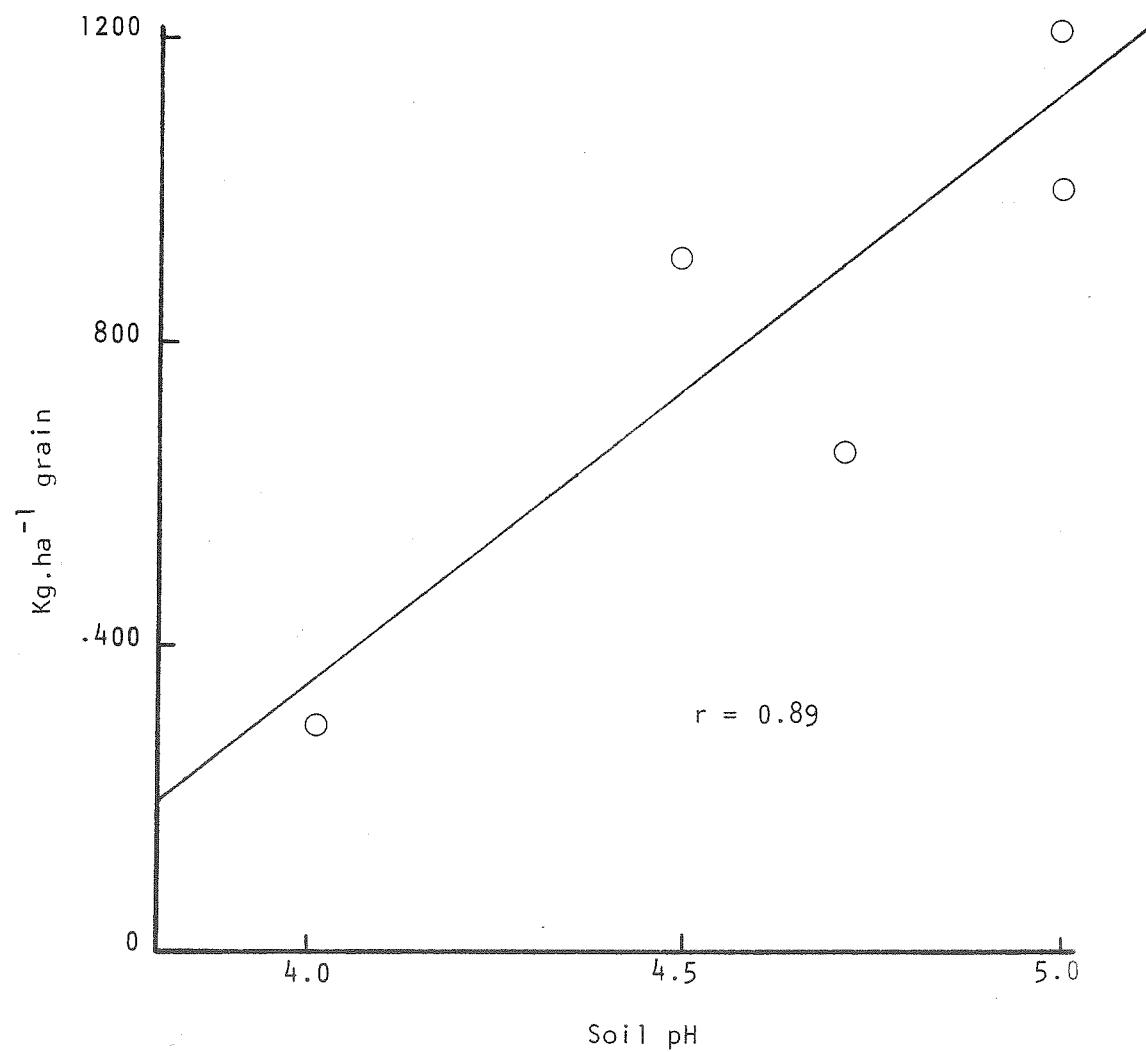


Fig. 9: Groundnut yield without lime in relation to soil pH.

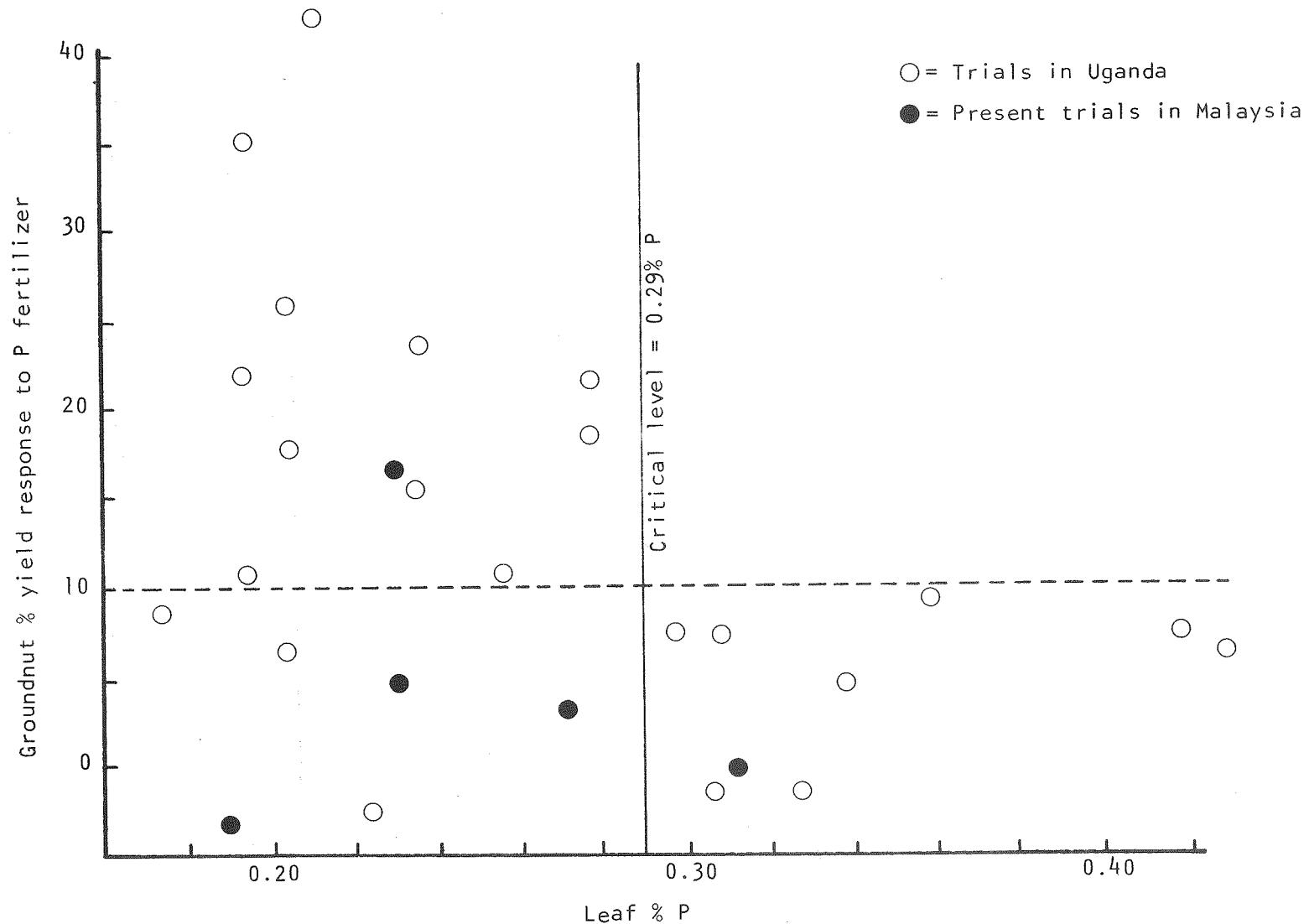


Fig. 10: Groundnut yield response to P fertilizer in relation to leaf P levels reached without P fertilizer.

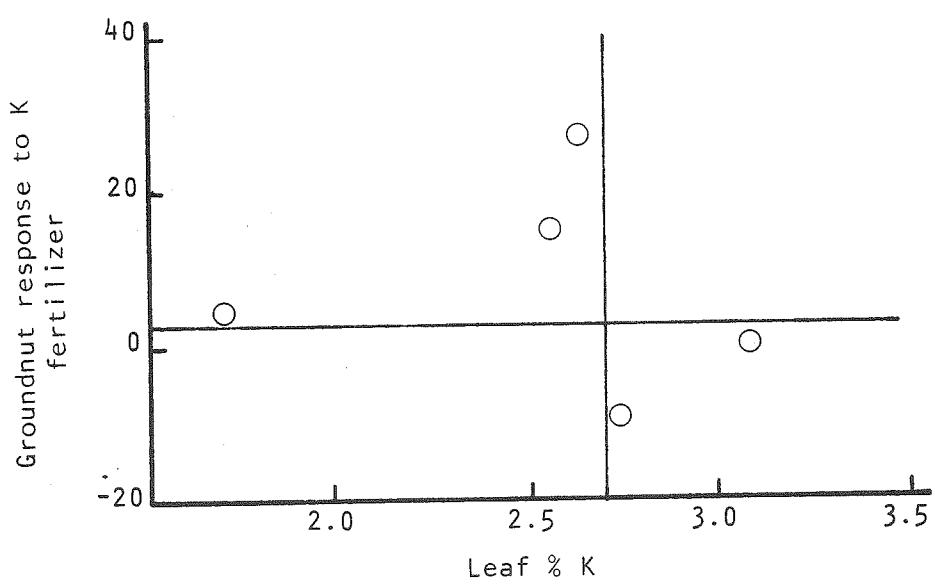


Fig. 11: Groundnut yield response to K fertilizer in relation to leaf K levels reached without K fertilizer.

## DISCUSSION

## SESSION 2

## 1. Interpretation of soil and leaf data for oil palm.

Mr. Hew Choy Kean asked Mr. Chan Kook Weng to elaborate on the integrated indices. Mr. Chan replied that the integrated approach requires the knowledge of the balance of elements in the plants. The indices show that the more unbalanced the leaf nutrient, the greater will be the difference between positive and negative index. A nutrient with large negative index will require more fertilizer applications to achieve balance with other nutrient.

## 2. Problems associated with leaf and soil analysis techniques in relation to cocoa.

Puan Siti Hajar Ahmad said that in page one of the paper it was mentioned that the age of leaf cannot be determined by the position on the branches or twigs. However, in page two it was mentioned that these two factors i.e. age and position were closely related. What is the relationship between these two and why position of leaf cannot be used to predict the age of leaf. Mr. K.C. Thong answered that by leaf age in relation to foliar analysis as a diagnostic aid is meant 'physiological' age. Because cocoa plant flushes at different times i.e. not all branches on the same tree or all trees, flush at the same time, to obtain a standard leaf for reproducible and meaningful leaf nutrient levels is very difficult since the physiological age of the leaf is not entirely or directly reflected by its position on the branch. This is one of the main reasons why foliar analysis is unreliable in the diagnosis of nutrient status of the cocoa tree.

3. Potential use of soil and leaf analyses data for groundnuts and other field crops in Malaysia.

Mr. Mohd. Khanif Yusop said that in Hawaii, Dr. R.L. Fox found good correlation between P uptake and P in solution and established 0.2 ppm in solution as the critical level. He asked whether this type of correlation exists under our condition and what is the critical level. Dr. Foster answered that they have not measured P in solution in fertilized soils yet but intended to do so. In unfertilized soils the P level in soil solution is generally too small to measure (<0.005 ppm).