soil and plant testing as a basis of fertilizer recommendations

PREFACE

The author of this paper, Dr. A. Cottenie, Professor of
Soil Chemistry at the State Agriculture University, Ghent,
Belgium, has wide international experience in matters of
soil testing and plant analysis, especially in tropical and
subtropical countries.

He has cooperated with FAO in the development of its soil
testing assistance programme since its inception in 1977.
In that same year, he took part in the Expert Consultation on
Soil and Plant Testing and Analysis which was organized by
FAO.[1] Based on the discussions at that Consultation,
Dr. Cottenie has prepared this paper which was first printed
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in the paper are those of the author and do not necessarily
reflect those of FAO.

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[1]   FAO Soils Bulletin 38/1. Soil and Plant Testing and
      Analysis. 250 p. 1980.

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I. INTRODUCTION

General principles of plant nutrition as formulated by Liebig and Mitscher-
lich, don't furnish the concrete information, necessary for practical
treatments in a given situation. In fact, we precisely need such concrete
information, for being able to apply the laws just mentioned. Thus, the
questions arise which parameters must be considered and how can they be de-
termined.

This leads to consider the concepts of nutrient diagnosis, as well as the
methodology and the corresponding equipment and instrumentation, for putting
it into practice.

The question may arise wether plant or soil analysis should be used for diag-
nosing crop nutrient requirements and making fertilizer recommendations.
The answer is that both methods may be complementary, not competitive and
the laboratories should be in the possibility to carry out both types of
analysis.

However it is recommendable to start soil analysis prior to plant testing.
The reason for this is that it is basically necessary to gather sufficient
knowledge of the soils which will receive the fertilizers, while plant tes-
ting may be carried out as well for observing the effect of fertilizer appli-
cations as for determining nutrient requirement of crops.

There are many possibilities and methods for testing soil chemical and nu-
trient properties. At each step one is faced with the problem of deciding
which factor to determine and which method to apply. Many publications report
the results of comparisons between different methods and, rather than repea-
ting such experiments, it is useful to stress the conclusions which are
generally in agreement and confirmed in different situations.

Soil testing has been independently organized in many countries and labora-
tories and this has resulted, through a history of trial and error, in the
development of a large number often only locally used methods. The figures
obtained in soil analysis are generally so much linked to the method being
used, that it is impossible to compare the results. Moreover, since these
results are "interpreted" on the basis of practical experimentation, it is
difficult and expensive to switch over to another method, even if this
should be a more attractive one (for reasons of reliability, etc.).

One advantage in setting up a new soil testing laboratory and making sugges-
tions for its planning is the possibility to take a start without being
linked by the above mentioned historical constraints.

It is indeed highly recommended to agree on a minimum of uniformity of methodology, which will favour the organization and practical installation and which will improve the reliability and confidence through the unique possibilities of comparing results, exchanging information and taking advantage of experience from elsewhere.

The question of standardization of soil analytical methods has been raised, but was not solved until now.

In planning a model for tropical soil and plant testing, the following questions have to be answered :

- which type of analysis or test should be carried out in order to diagnose the nutrient status of tropical soils and crops
- which type of experiment should be organized for calibration and as a basis for making interpretations
- which is the way to transform the experimental data into fertilizer recommendations.

In order to achieve these goals it is necessary to define appropriate methods, to establish corresponding and workable means and to outline the ways of using the latter.

1. The methods. There is certainly not one single method which might be considered satisfactory or the best one for determination of the nutrient status of the soil. Therefore a choice has to be made and the question must also be raised at which level this choice should be made. In some cases and for some elements there may not be one single method capable to cover the different soils and crops under consideration. Decisions concerning methods should be taken as a result of broader contacts and exchange of information, of coordination with other services and countries. It is felt that F.A.O. should take the lead to make suggestions and propositions for obtaining a certain harmonisation of methods on a regional basis. Therefore this booklet contains a choice of analytical methods, which have been applied in various conditions and which were judiciously selected. In some cases more than one alternative has been maintained in order to stimulate critical evaluation and to face the responsible soil chemist with the reality of practical soil testing. It should be stressed that the actual methods are not an end point in soil testing, even if a good harmonization on a large regional basis could be realised. Though improving and comparing methods is not the normal task of a plant and soil

testing service, its participation to programs of sample exchange and common comparative analysis is very useful.

2. <u>The working means</u>. Every analytical method can be put into practice in different ways and this is a question of available instrumentation and manpower as well as of volume and organization of the tasks. Even in case of a disconnection between developing methods and applying them in practice, it is necessary to organize their application as efficiently as possible is a function of the available means. Thus, running a soil and plant testing service is primarily a question of management, in view of improving the quality of the results, reducing the time between arrival of samples and delivery of results, saving labour and human effort and lowering prices. Such organization is of course different at each level of instrumentation. It is a general principle that every service tends continuously to increase and improve its possibilities and apparatus. The more this is realized, the more centralization is indicated, in view of an efficient and full-time use of heavier instruments and of their maintenance and servicing. The way in which human, instrumental and technical means are used determines the quantitative and qualitative possibilities of the service. At any level of sophistication, quality must be the leading preoccupation and the organization must contain a built-in system for controlling precision (reproducibility) and accuracy of the determinations. Several possibilities therefore exist e.g. incorporation of known standard samples and participation at international sample exchange.

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In 1971 CHAPMAN wrote "it is evident that as we look to the future, there will be an increasing need to use plant and soil analysis methods to guide and optimize fertilizer usage, to conserve natural resources and decrease or prevent pollution", and this view is largely confirmed to-day.

II. SOIL TESTING

1. General statements

In describing the nutrient status of soils the first step consists in deter-
mining field and laboratory factors characterizing the more general phy-
sical and chemical situation such as soil depth, tilth, slope, natural drai-
nage, stoniness, pH, electrical conductivity, humus content, cation exchange
capacity.

The knowledge of these factors permits to evaluate the soil under conside-
ration with regard to its basic aptitude for immediate cropping.

If for example the pH is too low or too high or if the content of soluble
salts is excessive, an appropriate treatment or management will be necessary
before normal fertilizer application can be recommended.

The following tests concern chemical and physico-chemical factors determi-
ning the soil fertility level or its content in available nutrient ele-
ments, for which a large variety of methods have been developed. Therefore
the choice of an analytical procedure for soil testing is more difficult
than for most other materials.

The fact that one has to make a choice between different possibilities,
which lead to different analytical results, has given rise to much dis-
cussion and controverse. The large number and the diversity of methods in-
dicate by itself their speculative character. As a result many countries
and laboratories have adopted different methods, which are difficult to
change when being applied on a routine scale.

In "Crops and Soils", February 1973, J. BENTON JONES published a paper
entitled "Should we or shouldn't we standardize soil testing" (6).

Having stated the lack of unanimity on this matter he concludes : "The
potential and changing role of soil testing demands standard or reference
test methods. The growing intrest in the environment and the concern about
overdosing our soils with fertilizer will demand more uniformly applied
test methods".

The proposal of models for soil testing provides a unique opportunity for
efficiency in establishing a type of techniques and equipment intended to
produce comparable and transferable results and information.

In this guide concrete proposals are made in order to promote soil testing in practice, without necessitating every single laboratory to make once again the considerable effort of comparing methods and to take the risk of an unhappy choice.

A too rigid standardization seems certainly unrealistic, but it must be emphasized that empirism should be discarded as much as possible. This means that newly created soil testing services, which are not yet engaged by a mass of data, obtained with formerly adapted methods, have the possibility to start with a selection of methods, being proposed to serve as common reference techniques, largely facilitating exchange of results and information.
It is not pretended that the selected methods are the best ones in every situation, but the advantages of more uniformity and standardization will undoubtedly represent a sufficient compensation.

Meanwhile, sufficient possibilities concerning practical operations and stepwize enlargement of activities, are present in order to stimulate further initiatives and to link routine activities with research. The latter should primarely aim to sustain the calibration and interpretation of soil analytical data.
Since soil testing is intended as a means for evaluating its fertility status, it is necessary to determine these variables and fractions of nutrient elements which are relevant with regard to crop response in terms of yield and uptake of elements. If a soil sample is successively treated with water and progressively agressive solutions, one can extract increasing quantities of each element, up to its total content (39).

The relatively small fraction of nutrients present in the soil solution is readily available. After its depletion by plant uptake, further supply must be provided by a sufficient replenishment, resulting from the transfer of elements stored in the solid towards the liquid phase.
Thus the distinction is often made between nutrient intensity I, indicating the quantities of elements present in a directly soluble form and nutrient capacity Q, giving the quantities which contribute to the replenishment of the soil solution as a consequence of desorption and solubilization. The ratio Q/I is a measure of the buffering capacity of the soil towards removal of an element from the soil solution.
Except for intensive vegetable cropping, where a permanent high nutrient intensity is required, determination of the nutrient capacity is more

meaningful for field crops, because it represents an estimation of the soil nutrient reserves which may become available during the growth period. In practice this determination is carried out by appropriate soil extraction.

The analysis of soil extracts must be completed by determination of the most important factors, influencing the storage possibilities and the availability of nutrient elements in the soil. Indeed, the behaviour and the uptake by the plant roots of fertilizer as well as native nutrient elements, are determined by the pH, humus and carbonate contents, cation exchange capacity, total nitrogen content, eventually oxidation-reduction conditions. Furthermore the ability of plants to absorb nutrients is dependent upon soil physical conditions, water supply and aeration, as well as upon soluble salt content of the soil solution.
The soil testing system, described in the following pages, is based on these considerations.

2. Analytical operations

At their arrival, the soil samples are identified and prepared for analysis by air drying, grinding and sieving. At this stage it is important to avoid contamination.
If necessary some physical determinations, such as texture-analysis, are carried out before chemical tests are started. The first series of chemical analysis comprise the so called direct determinations, which are performed on seperate aliquots of the sample. The determinations listed below, belong to the possibilities of the laboratory, but it is up to the agronomist to decide which ones must be carried out on a given soil sample.
- granulometric analysis
- soil pH (different modalities)
- conductivity and soluble salts
- content of free $CaCO_3$
- soil organic matter (oxidizable carbon)
- total nitrogen
- cation exchange capacity (C.E.C.)
- exchange acidity and lime requirement, eventually direct lime requirement determination
- gypsum requirement
- redox potential.

3. Determination of nutrient elements based on soil extraction

3.1. Principles and selection of methods

The first step of nutrient element determination in the soil is the separation of a fraction which is relevant with regard to crop nutrition. The methods being used with this aim are either intended for extracting one single element or for several elements simultaneously. Numerous comparisons were carried out in order to find an answer to the question which is the best method of analysing soils for available nutrient elements (1)(16)(24)(32)(37) As a result it was generally confirmed that no one single procedure is fitted for the different elements and soil conditions.

The mechanism of soil extraction is basically an equilibrium establishment between the solid phase of the soil and the liquid phase being the extracting solution. The phenomena by which elements pass into the liquid phase are solubilization, ion exchange and formation of soluble complexes. Special attention is to be given to the modalities of extraction, such as the soil/solution ratio, shaking time, pH etc.

Considering the fact that crop roots develop in a given volume of soil, which may have a different weight in function of its bulk density, there is a tendency to make soil extractions on a volume/volume basis. The importance of this remark is illustrated by the following example :

bulk density of soil	content expressed as	
	mg per kg soil	corresponding mg per dm^3 soil
1,5 (mineral soil)	100	150
0,2 (peat soil)	100	20

3.2. Extraction and determination of phosphorus

The mobilization of available phosphates is mainly a matter of solubilization. This is influenced by several factors such as pH and total acidity of the solvent, soil/solution ratio, complexing power of the solutes, contact time, temperature.

Unwanted interactions between soil and solvent resulting in pH change, as well as secondary precipitation reactions, must be avoided. For these reasons it is possible to select one appropriate method for neutral and calcareous soils, acid soils, lateritic soils , peat soils and possibly others.

The extracting method introduced by OLSEN e.a. has been widely used and tested, also in tropical soils (32).

It was initially proposed for calcarious soils, but proved also to be valid for neutral, acid and lateritic soils. Therefore Olsen's method is proposed as the most universal one.

In acid and lateritic soils available phosphates can also be determined by the method of BRAY and KURTZ (9).

Finally, much progress has recently been made with regard to simultaneous extraction of phosphorus and other nutrient elements.

The method of OLSEN which is suggested here for general application makes use of 0.5 n $NaHCO_3$ (pH 8.5) as an extractant.

The soil/solution ratio is 1/20 (original method weight/volume) and the shaking time 30 minutes.

The extracting solution is 0.5 n $NaHCO_3$ adjusted to pH 8.5 with NaOH and is designed to control the ionic activity of calcium, as a consequence of the excess of carbonate ions. Due to the high carbonate ion activity the Ca^{++} activity is decreased in function of the solubility product of $CaCO_3$. This has furthermore an indirect effect on phosphate activity. Indeed the decrease of Ca^{++} activity corresponds with an increased phosphate-ion activity in order to satisfy the solubility product of calciumphosphate. Thus a certain amount of the latter, if present as a phosphate nutrient reserve, is passing into solution.

3.3. Extraction and determination of nutrient cations

Summarizing an extended bibliography concerning the characterization of soils for nutrient cations, there is a general agreement to consider their exchangeable fractions. Especially in the case of potassium "there appears to be little justification for using any tests other than exchangeable + water soluble K for soil testing" (E.C. DOLL & R.E. LUCAS)(17), but also for Mg and Ca the same principle can be adopted.

Cation displacement with neutral normal ammonium acetate is generally adopted as a standard method. However, different modifications have been practiced for routine soil testing. The principle simplification consists in extracting by shaking instead of leaching the soil sample with the displacing solution.

3.4. Determination of available nitrogen

Though nitrogen is for many crops the most important fertilizer element, it is still not possible to propose a generally accepted and reliable method and soil testing for nitrogen is still a matter of more controverse than for any other nutrient. This is due to the different chemical forms of this element in the soil, their dependance and variability in function of (micro) biological activity and the narrow relationship between water and nitrate movement and uptake by plants. Field experimentation with nitrogen is obviously more necessary than with any other nutrient element.

More recently very encouraging results were obtained by determining mineral nitrogen (nitrates and ammonium) in soil profile samples taken before cropping or in early stages of growth (35).

This however includes much deeper soil sampling than traditional plow depth, especially for inorganic N analysis and for determination of soil water content. Such deep sampling is especially important in well drained and irrigated soils and makes possible to take the amounts of available nitrogen found within the whole root zone into account.

Special sampling devices are necessary if this method is to be applied and biological activity must be stopped or retarded in the collected soil samples. Therefore the samples must be immediately analyzed or refrigerated until analysis can be done.

Extraction of mineral nitrogen is carried out with a 1 n KCl solution and the analysis is made either by distillation or with specific electrodes. The C/N ratio is calculated from total C and N determinations. This value is to be considered in cases of high input of crude organic matter, possibly causing temporarily a high C/N ratio and a corresponding nitrogen deficiency.

3.5. Gypsum and sulfur requirement

- When soils contain an excess of exchangeable sodium, treatment with gypsum may lead to substitution of Na by Ca. In order to estimate the required amount of gypsum the soil is shaken with a saturated $CaSO_4$ solution. The quantity of calcium being retained by the soil as a consequence of the exchange with sodium (and some Mg) is used for calculating the gypsum requirement of the soil.

- The determination of available sulfate and eventual sulfur deficiency is based on extraction with water or different salt solutions. Monocalciumphosphate has been quite succesfully used for this aim and the level of extractable S in soil above which no response to fertilizer S would be expected is 7 to 12 ppm S (34). For the analytical determination of sulfate in the extracts, the turbidimetric method is used by most of the laboratories, but an interesting new colorimetric technique is also available.

3.6. Trace elements

Analysis of trace elements for diagnosing their presence and availability in the soil is generally not carried out on a routine scale, but when it is suspected that this might reveal the existance of a limiting factor of crop growth.
Though the possibilities for determining very low concentrations have been

much improved, the choice of appropriated extraction methods remain a difficult problem. Some trace elements such as iron, manganese, zinc, copper are mainly present as cations, while others like boron, molybdenum and selenium form generally anions. Varying fractions may be included in organo-mineral complexes, depending on pH, humus content, redox potential etc. There is a sufficiently good agreement concerning extraction of the following elements :

- Boron, which is generally extracted with hot water
- Molybdenum, most frequently extracted with neutral ammonium acetate.

In an analogous way more or less specific extractants have been proposed for the other biologically important trace elements. In the case of manganese fractionation of soluble, exchangeable, easily reducible and active forms has often been considered useful. Chelating agents, especially DTPA (*) are increasingly used for Zn, Fe, Mn and Cu. One of the most commonly used methods is those of LINDSAY and NORVELL (26).

Both potentially available quantities and mobile reserves of Zn, Fe, Mn and Cu can be determined by the method of LINDSAY and NORVELL (26), using 0.005 m DTPA, 0.01 ^{m} $CaCl_2$ and 0.1 ^{m} TEA (**) adjusted to pH 7,30 as an extractant.

Among other extracting methods for trace elements the following two are of interest :
- extraction with 0.5 n ammoniumacetate, containing 0.02 m EDTA at pH 4,65 (24)
- extraction with 0.5 n HNO_3

Especially with regard to trace elements, the interpretation system must be carefully worked out and amongst other factors include soil pH in its formulation.

The principle analytical techniques actually of generalized use for trace elements are :

(*) DTPA : diethylenetriaminepentaacetic acid
(**) TEA : triethanolamine

- atomic absorption : Fe, Mn, Zn, Cu, Co
                           Mo, Al, Hg
- spectrophotometry (colorimetry) : B, As
- spectrofluorimetry : Al
- specific electrodes : fluoride, nitrate.

Sufficient details for their application are given in appendix 1.
Recently a new possibility of emission spectrometry, making use of plasma
torch excitation, has been introduced, but this more expensive instrumen-
tation is apparently restricted to large laboratory and research units.

3.7. Simultaneous extraction

SOLTANPOUR & SCHWAB (36) made an interesting proposal to develop a single
extracting method, combining the characteristics of the following original
separate solutions used for phosphorus, potassium, nitrates and trace
elements :
- bicarbonate for the extraction of phosphorus
- ammonium for the extraction of potassium
- DTPA for chelation of trace elements
- water for nitrates.
This new extracting solution is 1 m NH_4HCO_3 at pH of 7.6 and contains
0.005 m DTPA (diethylene triamine pentaacetic acid).
The published results show a high correlation with Olsen's P test, ammonium-
acetate K test and Lindsay and Norvell's DTPA-Zn, Fe, Cu and Mn test.
In view of the simplification which might be obtained with this procedure,
it is also given in sufficient detail (Appendix 1).
MEHLICH (29) also developed a new extractant to meet"the need for extracting
representative portions of the largest number of plant available nutrients
in a single extract over a wide range of soil properties". Its composition
is 0.2 n NH_4Cl - 0.2 n HOAc - (0.015 n NH_4F - 0.012 n HCl) at pH 2.5 fa-
vouring the effective extraction of exchangeable cations, rock phosphate
and other calcium phosphate forms in calcareous soils, as well as alumi-
nium and iron forms of phosphorus.

4. Recording and presentation of results

For a smooth run of laboratory activities it is necessary to record every result and information in a systematic and standardized way. To do so appropriate forms must be used, which are so conceived that the operator is being guided in a step by step procedure.
This necessitates different types of work-sheet forms :

4.1. Sample entrance form

Information sheet, giving origin of the sample, date, general and particular characteristics and remarks concerning local soil situation and crops. On this form all important soil properties which are not recognisable in the soil sample should be recorded, such as soil depth, slope, stoniness, drainage and past cropping history (see appendix).

4.2. Internal documents

During their way through the laboratory and the interpretation office, the samples are accompanied by the following types of documents :

a.- Analytical forms : In view of efficiency the samples are grouped in series of 24 (or another convenient number). For each such series the results are noticed on one form per element.

b.- Sample result forms: When all required determinations are carried out on one series, the results of each individual sample are brought together on one form, containing also the general characteristics of that sample. These forms are checked by the laboratory supervisor before they are handed to the agronomist for interpretation.

It is desirable that both groups of forms have a different color e.g. white, pink and yellow. Colors to be avoided are red, green and bleu.

4.3. Soil test report for external use

The final soil test report contains three parts, respectively giving :
- information concerning the field, sampling, cropping
- analytical data and judgment
- fertilizer recommendations.

This document is destined to the extension officer and the farmer. It should not be too complicated, but remain clear and written in terms that are sufficiently comprehensive for the practician.

Examples for possible presentation of each part are given below.

4.4. Further practical recommendations :

- Amounts of nutrients are preferably expressed as milligrams, grams or
  kilograms of the element : e.g. (milli)gram N, K, P, Mg
- Concentrations in the soil are most efficiently expressed as ppm (parts
  per million), this is milligram per kg (weight/weight). However there is
  a tendency to express the results also as milligram per dm^3 (weight/
  volume).

All forms should contain unequivocal indications and column headings.
Especially the units in which results are expressed should be clearly
indicated.

In order to assure a future maximum use of all produced data, it is advi-
sable to standardize from the start a record keeping system fitted for
later expansion. This concerns the final storage of results and information.

Remark 1

Expressing soil test results on a uniform basis is highly recommended in
order to favour comparison of results and information.
Recognizing that the soil:root association is a volume relationship, many
soil chemists tend actually to express soil test results on a weight/
volume basis, as already mentioned.
When the bulk density of the soil is BD, ten mg/dm^3 = mg/kg x BD and
the following list may be useful.

Bulk density	mg/dm^3 corres-ponding with 100 mg/kg soil	mg/kg corres-ponding with 100 mg/dm^3 soil
1.5	150	66,6
1.4	140	71,4
1.3	130	76,9
1.2	120	83,3
1.1	110	90,9
1.0	100	100,0
0.9	90	111,1
0.8	80	125,0
0.7	70	142,9
0.6	60	166,7
0.5	50	200,0
0.4	40	250,0
0.3	30	333,3
0.2	20	500,0
0.1	10	1000,0

Remark 2

In the case of major nutrient cations, it may be useful to express their
values in milli-equivalents in order to fit them in a balance system
where the cation exchange capacity is expressed in milli-eq per 100 g soil.
Indeed, quantities of elements may only be added and ratios may only be
calculated if they are expressed in terms of (milli-)equivalents.
The equivalent amount of an element is calculated by dividing the number
of (milli)grams by its "equivalent weight".
The equivalent weight is the atomic weight devided by the valency number.

Examples :

N : atomic weight = 14,00
   valency number = 1
   equivalent weight = 14,00

P : atomic weight = 31,97
   valency number = 3
   equivalent weight = $\frac{31,97}{3}$ = 10,32

Ca : atomic weight = 40,08
   valency number = 2
   equivalent weight = 20,04

K : atomic weight = 39,10
   valency number = 1
   equivalent weight = 39,10

Some useful conversion factors are listed below :

Element	atomic weight	equivalent weight of the element	the oxide
N	14,00	14,00	–
P	31,97	10,32	23,66 (P_2O_5)
S	32,06	16,03	–
Ca	40,08	20,04	28,04 (CaO)
Mg	24,31	12,15	20,15 (MgO)
K	39,10	39,10	47,10 (K_2O)
Na	22,99	22,99	30,99 (Na_2O)

5. Interpretation of results

The value of numerical results of soil testing depends upon the use one can make of them and their full exploitation requires an input of several other types of information, such as physical environmental characteristics, hydraulic data, crop specifications, management, etc. It should be stressed that soil test results are a measure of available nutrient contents but do not indicate the addition needed to produce a given yield increase on a deficient soil. The main problem is to predict probable crop response to fertilizer applications and this is largely dependent upon local circumstances and concrete field conditions.

Yield increase by fertilization is of course more probable in regions with sufficient rainfall, spread over the whole vegetation period than in unfavourable physical conditions as is often the case in the tropics. Therefore interpretation of soil analysis is more difficult in tropical than in moderate climatic conditions. A working guide for data treatment and fertilizer recommendations should be actualized and periodically up-dated. Examples of guides are published by several U.S.A. soil testing laboratories and agricultural experiment stations (*).

In order to make fertilizer recommendations it is necessary to relate the soil test values to the rate of application of the nutrient required for optimum yield. To do this yield has to be related to soil test values and to fertilizer application and this is a question of field observation and experimentation.
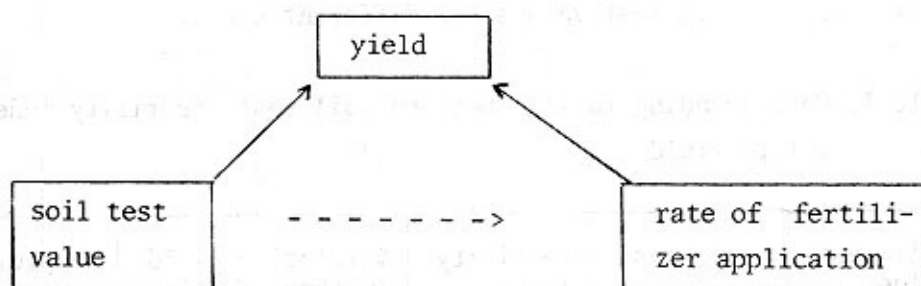
(*) - Guide to fertilizer recommendations in Colorado-Soil analysis and computer process (Colorado State University - Fort Collins - USA)

- Crop fertilization based on N.C. Soil Tests (Soil testing laboratory, Agronomic division, North Caroline Department of Agriculture, Raleigh, USA)

- Fertilizer recommendations and computer program key (Agricultural experiment station, Auburn University, Auburn, Alabama, USA).

Interpretation of soil test results may proceed in different steps as follows :

5.1. Classification of soil nutrient element levels

For each element the extracted amounts range between limits as listed in tables 1 and 2, where they are grouped in 5 classes. Such nutrient level ranges are only valid for the particular extraction method used. In the case of nutrient cations, the cation exchange capacity (CEC) is an important factor with regard to the quantities of elements present.

On the basis of these reference tables one can conclude if the soil is poor, medium or rich in a given element, independently from the reaction of a given crop towards this situation. Thus an eventual limiting factor may be identified in case of a very low figure, without strictly necessitating any further biological test.

Soil test ratings may be linked to a fertility index, giving the percent sufficiency of the nutrient status with regard to expected yields, as proposed by COPE and ROUSE (14).
If the yield obtained without fertilization represents 50 % of the opti - mum, the fertility index is denoted 50. In the same way an expected maximum yield (100 %) corresponds with a fertility index of 100, but if the fertility index is increasing further, expected yields will no more be higher (Table 3). It is clear that these fertility indexes are unequally related to the soil test values for different crops.

Table 3. Corresponding ratings between soil test, fertility index and relative yield

soil test value	soil test class	fertility index	% relative yield (without ferti- lisation)	expected crop response
variable	very low	0-50	<50	definite
scale in	low	50-80	50-80	probable
function	medium	80-110	80-100	less likely
of crop	high	110-200	100	unlikely
	very high	200-400	100	unlikely

Table 1. Suggested interpretation of phosphorus nutrient levels in mg P per kg soil (ppm)

Class	Olsen's extraction	Simultaneous extraction (SOL-TANPOUR & SCHWABB)
very high	>25	>12
high	18 - 25	8-11
medium	10 - 17	5-7
low	5 - 9	2-5
very low	<5	<2

Table 2. K, Mg, Ca per kg soil (ppm)

Texture	CEC in meq/ 100 g	Qualification	K	Mg	Ca
low CEC	± 5	very high	>100	>60	>800
		high	60 - 100	25 - 60	500 - 800
		medium	30 - 60	10 - 25	200 - 500
		low	15 - 30	5 - 10	100 - 200
		very low	<15	<5	<100
medium CEC	± 15	very high	>300	>180	>2400
		high	175 - 300	80 - 180	1600 - 2400
		medium	100 - 175	40 - 80	1000 - 1600
		low	50 - 100	20 - 40	500 - 1000
		very low	<50	<20	<500
high CEC	± 25	very high	>500	>300	>4000
		high	300 - 500	120 - 300	3000 - 4000
		medium	150 - 300	60 - 120	2000 - 3000
		low	75 - 150	30 - 60	1000 - 2000
		very low	<75	<30	<1000

Remark : The figures mentioned in these tables are given as guidelines, but will not be valid under all circomstances as a definite qualification.

5.2. Facing soil test results with crop response

Different crop groups have varying nutrient requirements and don't react in the same way towards the nutrient levels of the soil. Therefore it is helpful to distinguish crop groups with regard to their specific nutrient requirements e.g. crops with low, moderate or high P, respectively K requirements.

The total requirement has been defined as the total amount of an element present in a crop and needed for optimum crop production. Potatoes, sugar beets, alfalfa need high phosphorus and potassium rates, while cereals, pasture crops, beans have much lower requirements for these elements that fertilization of irrigated crops must be much higher than on dryland. Since the production level is also largely influenced by management practices and nitrogen application, the total uptake and crop removal of P, K, Ca, Mg is a function of these factors, which are linked to a practical yield goal.

5.3. Fertilizer recommendations

Fertilizer recommendations are finally formulated on the basis of expected crop response in the local ecological situation, plant requirement and economic conditions.

This final step, which is the most difficult one, needs sufficient information with regard to crop behaviour in practical field conditions, where typical factors, which cannot be observed nor measured in the laboratory, are also acting.

Furthermore at this stage it is possible to introduce corrections, judged necessary in function of some interactions, which were not yet regarded when considering the nutrient elements as single values. Indeed, interpretation of soil testing results consists also in combining different factors with their relative weight, as discussed in the following paragraphs. Guidelines for fertilizer recommendations of different crops may be conceived as follows :

Soil test result	Fertilizer recommendation in kg per Ha $\begin{cases} P \text{ or } P_2O_5 \\ K \text{ or } K_2O \end{cases}$			
	crop A	crop B	crop C	. . . .
very low	40	60	80	. .
low	30	40	60	. .
medium	20	20	40	. .
high	10	10	20	. .
very high	0	0	0	. .

Fertilizer recommendations and information processing may be carried out with the aid of computer techniques. When an increasing amount of information is available, the use of a computer is a logical step to include every useful acting factor. Programming for the computer requires a good knowledge of all aspects of fertilization and crop nutrition being considered : crop requirement, leaching, possible fixation , role of soil organic matter, etc.

5.3.1. Basic cation saturation ratio concept (BCSR)

This principle consists in comparing the actual exchangeable bases with the values considered as optimal. In a general way Mc LEAN (29) stated that ranges from 65 to 85 % Ca, 6 to 12 % Mg and 2 to 5 % would be quite satisfying. From the CEC-determination of the tested soil and its content in exchangeable basis, the corrective treatment can be calculated in order to bring the cation nutrient situation nearer to the optimal one. This method called BCSR, is primaraly fitted **for** exhausted or highly weathered soils, as well as for grassland where a high proportion of Mg is required in view of a satisfying animal nutrition.

It is practical to consider the following 3 CEC soil groups :

light : <10 milli-equiv. per 100 g soil

medium : 10-20 milli-equiv. per 100 g soil

heavy : >20 milli-equiv. per 100 g soil

For knowing the base saturation the exchangeable cations Ca, K and Mg are expressed as milli-equivalents per 100 g soil, totalized and expressed as percent of CEC.

For soil classification purposes the base saturation classes are defined as follows :

<35 % unsaturated

35-80 % moderately saturated

>80-85 % saturated

MEHLICH (29) distinguishes the following situations :

% Ca saturation	% base saturation	deficiency designation
<35	<45	severe
36-55	46-65	poor to moderate
56-70	66-85	optimum for acid tolerant plants
>70	>85	optimum for acid intolerant plants

5.3.2. K/Mg ratio

The ratio between K and Mg is an important factor with regard to possible induced magnesium deficiency.

Once again it must be stressed that calculation of the K/Mg ratio needs the expression of these elements in milli-equivalents, and 12 milligrams Mg are equivalent with 39 milligrams K. When the K/Mg ratio on an equivalent basis is higher than 1 the Mg situation of the soil must be carefully watched. On a weight basis this is a K/Mg of 3,25.

6. Calibration of soil tests

When a soil testing method is put into practice the laboratory tests must be related to crop responses in function of fertilizer rates. At this moment there is already a large amount of information available, giving critical and response levels, fertility classes and corresponding crop behaviour as observed in different ecological conditions.
This information has been obtained by means of numerous field experiments over a long years period. It is of course indicated to take as much profit as possible of it and here rises the question of the necessity to repeat such experiments in every country and to which extend.
As a matter of fact, there is a general agreement concerning the necessity of field experiments in view of linking together analytical results and fertilizer effects. This has been confirmed and repeated by many soil and fertilizer specialists as illustrated by the following citations :

. "A good soil testing program must accompany a good field experimental program if the soil tests are to mean anything" (F. VIETS, 1977) (39)
. "Without the results of field experiments the recommendations of soil testing services will contain a very large element of guesswork" (G.W. ARNOTT, 1977) (2)
. "Experiments for calibration of chemical tests that provide the basis for recommendations to farmers must be conducted in the field" (J.J. HANWAY, 1973) (21)
. "Soil tests are calibrated by correlating them with the yield results of field experiments" ... "The establishment of a soil testing service, including the preparatory field work and research involved in it, is not a matter of one or two years but longer and should be considered as the first period of a continuous and gradually improving service for the similarly improving farm operations in an area or country" (G.F. HAUSER, 1973) (22).

These and many other statements unanimously indicate the necessity to carry out field experiments in the environmental conditions where soil and plant testing will be used for fertilizer recommendations.
However, while "such experimentation is not easy, and it is not cheap, but there is no adequate alternative" (J.J. HANWAY, 1973), pot experiments may be organized to provide usefull information to the soil testing service.

6.1. Pot experiments

The aim of pot experiments is double :
a) to compare crop response and evolution of analytical indexes towards uniform treatments of soils taken from different areas.
b) as a screening method to select the soils and places where field experiments can be located with a maximum of chances for significant response towards a given nutrient element.

Among the numerous alternative possibilities the so called substractive method of CHAMINADE (11) presents several advantages and it has given excellent results in tropical conditions. The original method makes use of small containers in which 1 kg soil is grown with rye-grass or a tropical grass species as a test plant. The yield of successive cuttings is determined in function of a complete fertilization and in absence of one nutrient element. So the following 7 treatments may be applied :

Complete fertilization : N, P, K, Ca, Mg, S, trace elements
same without P
same without K
same without Ca
same without Mg
same without S
same without trace elements

The yields are expressed in % of the one obtained with complete fertilization and these yield-indexes give the following informations :
- the nature of any observed deficiency
- the comparative importance of these deficiences
- the evolution and exhaustion of the soil nutrient reserves.

6.2. Field trials

As already mentioned, the soil testing service must not restrict its activities to laboratory analysis and pot experiments, but should also organize field trials for calibration of the analytical system, on major soils of the area served by it, for improving fertilizer recommendations.
After having stated the probability of positive response by means of a pot experiment, the field trial is the most indicated way to verify the effectiveness of a fertilizer application on a given soil and for a given crop. Thus the field experiment is normally preceeded by analyses and pot experiments having furnished sufficient indications for expecting a sig-

nificant effect of treatments. At this stage "relative yield data cannot
be used effectively even if they correlate better with soil test than
the absolute crop responses. The reason is obvious. Fertilizer advice is
based on economic considerations. A certain percent yield increase may
be a high or a low absolute amount and it is therefore no basis for the
required benefit calculation. This shortcoming cannot be compensated by a
somewhat better correlation" (HAUSER, 1973)[22].

Concept and layout of field experiments for observing crop responses to
different fertilizer dosis should be relatively simple and an excellent
method has been described by G.F. HAUSER [22].   The proposed treatments
are simply five rates of the tested element equally spaced, the lowest
being zero and the highest so chosen to obtain a maximum yield. In addi-
tion to these increasing rates of the tested nutrient all plots should
be given a basal dressing of the other main nutrients. If trace element
deficiencies have been observed in the area these nutrients should be
applied too in suitable quantities.

The experiment shows the yield response of the test crop in function of an
element under study in the local conditions. It should be recommended to
apply the same scheme for convenience of organization and comparison.
A primary yield versus dosis graph is obtained which represents an addi-
tional information to the interpretation scale belonging to the diagnosis
method.
When more such experiments are progressively carried out on soils of dif-
ferent nutrient levels, a family of crop response curves is obtained :
one for each dosis in function of the soil test index. The general aspect
of such curves is illustrated in graph 1 and full details of their mani-
pulation are described by G.F. HAUSER [22].

Fig. 1.
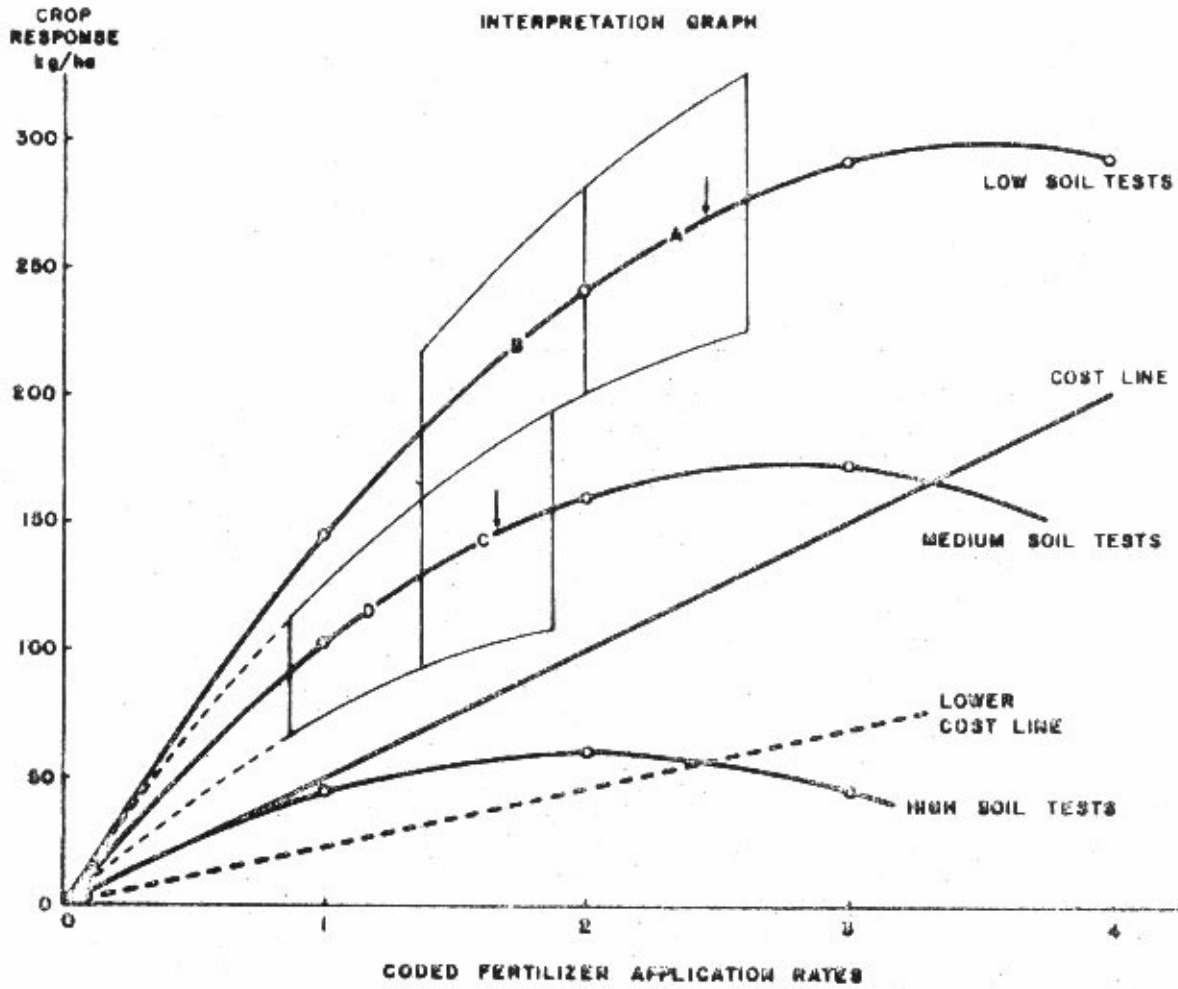
(from G.F. HAUSER, 1973. The calibration of soil tests for fertilizer recommendations; F.A.O. Soils Bulletin, 18).

6.3. Sufficiency levels of available nutrients concept (SLAN)

Another procedure which can be recommended is the GATE and NELSON graphical method, plotting soil test results versus percentage yield (10).
The resulting scatter diagram is divided into four quadrants, maximizing the number of points in the positive quadrants while minimizing the number of points in the negative ones.

% yield
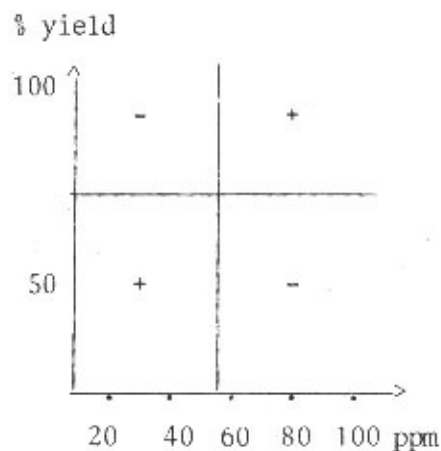
The soil test value separating the two groups was called the "critical level" signifying that the probability of response to fertilizer is large below this value and small for the points of the other group.
The following examples are taken from NELSON & ANDERSON (31) and show the critical levels for soil P as extracted by Olsen's method respectively valid for wheat and potatoes in Bolivian soils.
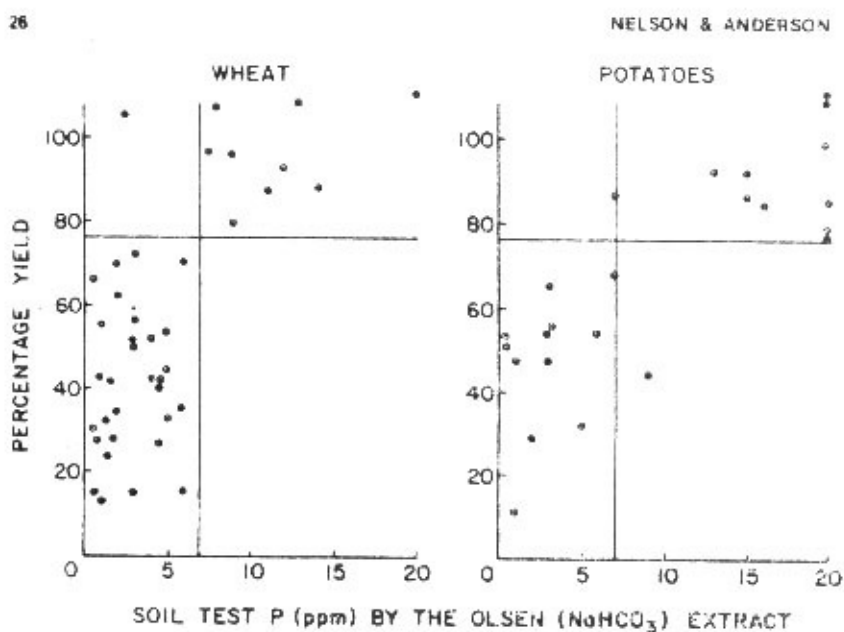
Figure 3. Scatter diagrams of percentage yield of wheat and potatoes versus soil test P by Olsen method for Bolivian data. International Soil Fertility Evaluation and Improvement Program, 1972.

III. PLANT TESTING

1. Conditions and possibilities

In plant testing there is less choice for analytical methods than in soil
testing since plant samples are analyzed for total mineral element con-
tents. The main problems in plant testing are sampling and interpretation
of results. The complex relationship between plant tissue contents of
mineral elements and the nutrient status of the growth medium has been
discussed in many publications. In spite of direct or indirect influences
of many factors in relation to soil physical and chemical properties, to
plant species and even clones, to season and physiological age, there are
sufficient exemples of succesful application of plant testing with the
purpose of nutrient diagnosis.

Despite such encouraging experiences, soil testing generally continues to
precede plant testing for routine fertilizer advisory purposes.
However, every laboratory for soil analysis normally has sufficient fa-
cilities for plant analysis as well, and the latter makes it possible to ob-
serve the effect of experimental treatments and to verify the uptake of
fertilizer elements.
Thus plant analysis is recommended in the following order of priority
and applicability :

1.- To support experimental work and to verify the effect of treatments in
    pot and field trials. Such analysis give supplementory information
    concerning interactions and antagonisms among elements.
2.- To elucidate suspected deficiencies, suggest additional tests and
    locate areas which are exposed to specific nutrient troubles.
3.- To serve as a criterion for testing the quality of certain crops such
    as forage and vegetables.
4.- To serve as a tool in systematic nutrient diagnosis and fertilizer
    guide for a particular crop or group of crops.

The item 4 is to be considered after serious and extensive preparation and
can initially be conceived to support soil testing.
For several reasons, plant testing is most likely to be succesful with
perennial crops. The first reason is that perennials are remaining witnes-
ses of their ecological and nutrient environment, while the information
obtained with annual species comes usually too late for an effective in-
tervention on the same crop.

Another reason is that most of the available information concerns the important perennial crops. The best results have generally been obtained with perennial crops under tropical and subtropical climate. Thus useful data are available concerning citrus, olives, banana, oil palm, rubber, coffee, cocoa, coconut, papaya, pine-apple, as well as cotton, cassava etc. (Table 5).

2. Factors influencing the mineral element composition of plants

There are many factors influencing indirectly the mineral element content of plants, which finally is the resultant of all acting parameters.

2.1. Soil parameters, such as texture, cation exchange capacity, humus content, soil density and aeration, oxidation-reduction potential and pH, all contribute to determine the availability of nutrient elements. This list could be extended to climatic and meteorological factors such as rainfall, temperature, light, which influence soil humidity.

2.2. Plant species behave in a more or less caracteristic way and this is clearly illustrated by the varying mineral composition of different plants growing together in the same soil or substrate.
The following observations have been generally confirmed : dicotyle plants contain more Ca, Mg and B than monocotyles, the latter showing higher levels of K.
Crucifers tend to accumulate sulfur, while rice, oats and spinach are known to be relatively rich in Fe. Sodium is quite easily accumulated by beets, rye, spinach, cotton, date palm, but remains at low level in maize, potatoe and sunflower.

2.3. Physiological age and part of the plant to be sampled

During the early vegetation period, the rate of nutrient uptake is high and this consequently leads to high nutrient contents in the plant tissues. Increasing production of organic matter is responsible for a dilution effect in the middle of the vegetation cycle, corresponding with decreasing nutrient concentrations. This phenomenon is most pronounced with regard to NO_3^--nitrogen.

Thus physiological age is an important factor of variability and young, metabolically active leaves generally contain higher amounts of nutrient elements. Accumulation of proteins corresponds with higher levels of N and P and several observations confirm that the highest P and N contents are found in cereals at the tillering phase.

During further growth phosphorus contents decrease generally less than N and K, the latter being very mobile and even being partly returned to the soil at the end of the growth period of several crops. On the other hand, aging of plants may also correspond with increased contents of some elements such as Ca and Mg.

Different parts or tissues of the plants also contain and accumulate varying amounts of elements and this is of course important with regard to the choice of the plant part to be analysed, which should be the best "index part".

Fruits generally contain small amounts of mineral elements, because they mainly act as stores for organic matter, such as carbohydrates or lipids.

3. Practical aspects of applied plant analysis

Different authors have reviewed and compiled a large part of the available information concerning nutrient diagnosis using plant analysis.
GOODALL and GREGORY (20) were the first to compile a large number of data and their pioneer work was later completed by CHAPMAN (12, 13). Extensive tables of analytical values were recently also published by BERGMANN & NEUBERT (7). In principe the concentration ranges are split up in 5 levels, corresponding respectively with deficiency symptoms, low range, intermediate, high and toxic levels.

It is impossible to review or to summarize the numerous publications on particular applications and problems related with plant testing for nutrient requirement evaluation. Locally employed techniques and experiences are not always conclusive nor uniform. WALSH & BEATON's book (1974) "Soil testing and Plant Analysis" contains ten chapters, treating separately the methodology for sugar beets, sugarcane, cotton, soybeans and peanuts, small grains, corn and grain sorghum, vegetable crops, orchards, forage crops and forests, each of them written by specialists of the matter.

The best results have generally been obtained with perennial crops in the mediterranian to tropical countries. Thus useful information is available concerning grapes, citrus crops, olives, banana, oil palm, rubber, cotton, papaw etc.

4. Sampling and pre-treatment of samples

Sampling is of course the first important step and it is necessary to standardize plant or leaf sampling techniques as perfectly as possible. Rigid observation of precise indications is the first step of any plant testing system. The general rule is to sample upper recently mature leaves and the recommended time for sampling is just prior to the beginning of the reproductive stage for many plants. When nutrient disorders are suspected, sampling may be done at the time at which the symptoms are observed. It is not possible to give more detailed instructions in this report, but these can be found in specialized publications (5)(8)(27).

It is essential that the laboratory provides full instructions for this important step and that the sample taker has the necessary equipment for proper cutting, cleaning, packing and eventually mailing the samples, as well as for labelling them and completing the necessary forms.

Pre-treatment of samples comprises eventually cleaning, drying, grinding and storing and this also must be carried out without improvisation, but in a systematically organized way.

MARTIN-PREVEL (27) reported international cooperation in view of improving sampling methods and foliar diagnosis of banana, and grouping nearly all scientists working in this field.

It is clear that the sampling procedure of banana leaves, with surfaces of 1 to 2 m^2, constitutes an important parameter of the method.

Oil-palm proved to be an ideal crop for foliar diagnosis and its well defined phyllotaxis permits an easy standardisation of leaf sampling.

5. Analytical problems with regard to plant testing

5.1. Destruction of organic matter

The first step in the analysis of plant samples is the destruction of organic matter in order to obtain a solution of inorganic ions.
This may be achieved by dry ashing or by wet digestion. Dry ashing is not

recommended for plant material high in silicon content.
- If dry ashing is preferred, the following remarks must be kept in mind :
  . simple ashing of 1 g dry matter at 450°C and dissolving in HNO_3 or
    HCl permits the determination of P, K, Mg, Ca, Na but part of Fe, Mn,
    Zn, Cu, etc. may be unsolubilized as silicates while ashing
  . an additional step consisting in a treatment of the unsoluble ash
    with hydrofluorhydric acid (HF) is recommended for the determination
    of trace elements. In this way silicium is volatilized as SiF_4 and the
    second ash solution is joined to the first.
- If wet destruction is carried out the dry matter is treated on a hot-
  plate with conc. H_2SO_4 and small additions of H_2O_2. This technique is lon-
  ger but easier to perform on a larger scale, especially when a destruction
  rack is available. Moreover nitrogen analysis, which requires a special
  destruction when the samples are dry ashed, can be made in the same digest.
- In any case sulphate determination is only possible after a special des-
  truction in the presence of $Mg(NO_3)_2$.

5.2. Analytical methods

The analytical methods for plant ash solutions or digests are fundamentally
the same as for soil extracts. The techniques are volumetry, spectrometry
(colorimetry), potentiometry, flame photometry and atomic absorption.

5.3. Precision and accuracy

In order to give reliable results the differences observed in function of
varying nutritional status must be large enough to overcome the natural
variability and the inevitable sampling errors.
The question which total error is acceptable in view of demonstrating a
significant difference δ between two samples can be answered by calculating
the maximum allowable standard error σ from the formula :

$$\sigma \leqslant (\frac{n}{2})^{\frac{1}{2}} . \delta . (t_1 + t_2)^{-1}$$

where t_1 and t_2 are the critical t values of the Student-distribution and
n the number of replications (15).

Table 4 contains the calculated maximum standard errors σ at the levels
p = 0.05 and p = 0.01, for 3 chosen probabilities P 0.80, 0.90 and 0.95
and with respectively 2 and 3 replicates.
The same data may be graphically represented so that a continuous scale
is obtained (fig. 2). This graph makes possible to judge about the relia-
bility of the observed differences.

6. Interpretation

The relationship between nutrient concentration in plant tissue and crop
behaviour has been described as follows (19) :

Acute deficiency	Latent deficiency	optimal nutrient status	luxury consumption	Excess or toxicity
Visual symp-toms Direct ef-fect of fer-tilization or leaf application	No visual symptoms Better yield and quality by fertili-zation	Good growth and gene-rally good quality	Good growth but internal accumulation Possible in-teractions	Yield decrease Possibly visual symptoms

limit of   limit of   start level
visual   yield   of toxicity
symptoms   response
         (critical level)

The situations to be distinguished are :
- low concentration : indicative for serious deficiency and sharp reduc-
  tion of yield. The term critical concentration is used to indicate the
  level below which this occurs.
- optimum level : the concentration range corresponding with sufficient
  nutrition.
- high concentration : enrichment which may be due to high nutrient level
  in the soil or to reduced growth. Indeed the observed concentration of
  an element is the ratio between the amount taken up and the already pro-
  duced plant mass. If growth is restricted by another limiting factor,
  the concentration will be relatively high in spite of low uptake of the
  considered element.

Table 4. Maximum standard errors σ permitting to confirm a difference δ at significance levels $p_1 = 0.05$ and $p_1 = 0.01$ (2 replicates) (*)

	$p_1 = 0.05$			$p_1 = 0.01$		
Probability . . .	0.80	0.90	0.95	0.80	0.90	0.95
Difference δ						
1.000	251.2	208.1	171.2	124.6	113.0	101.2
5.000	1.256	1.040	856.2	623.0	564.9	505.8
250	62.80	52.02	42.81	31.15	28.24	25.29
500	125.6	104.0	85.62	62.30	56.49	50.58
25	6.280	5.202	4.281	3.115	2.824	2.529
50	12.56	10.40	8.561	6.230	5.649	5.058
10	2.512	2.081	1.712	1.246	1.130	1.011
20	5.024	4.161	3.425	2.492	2.260	2.023
5	1.256	1.040	0.856	0.623	0.564	0.506
10	2.512	2.081	1.712	1.246	1.130	1.011
2	0.502	0.416	0.342	0.249	0.226	0.202
4	1.005	0.832	0.685	0.498	0.452	0.405
1	0.251	0.208	0.171	0.125	0.113	0.101
2	0.502	0.416	0.342	0.249	0.226	0.202
0.1	0.025	0.021	0.017	0.012	0.011	0.010
0.5	0.126	0.104	0.086	0.062	0.056	0.051

(*) All values of σ are calculated with 4 figures.

Fig. 2. Maximum standard deviation σ for observing a significant difference δ

level $p_1 = 0.05$

level $p_1 = 0.01$

difference σ to be detected

difference σ to be detected
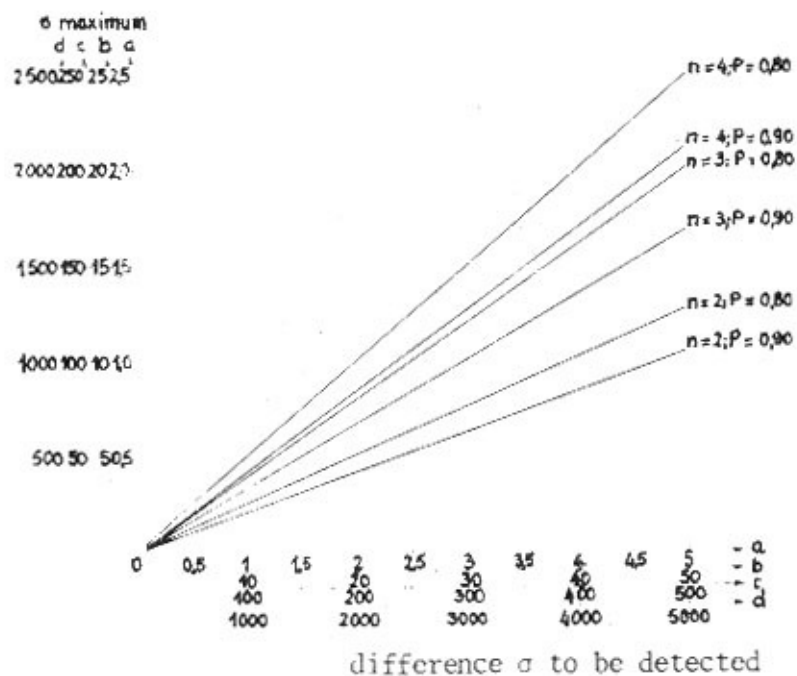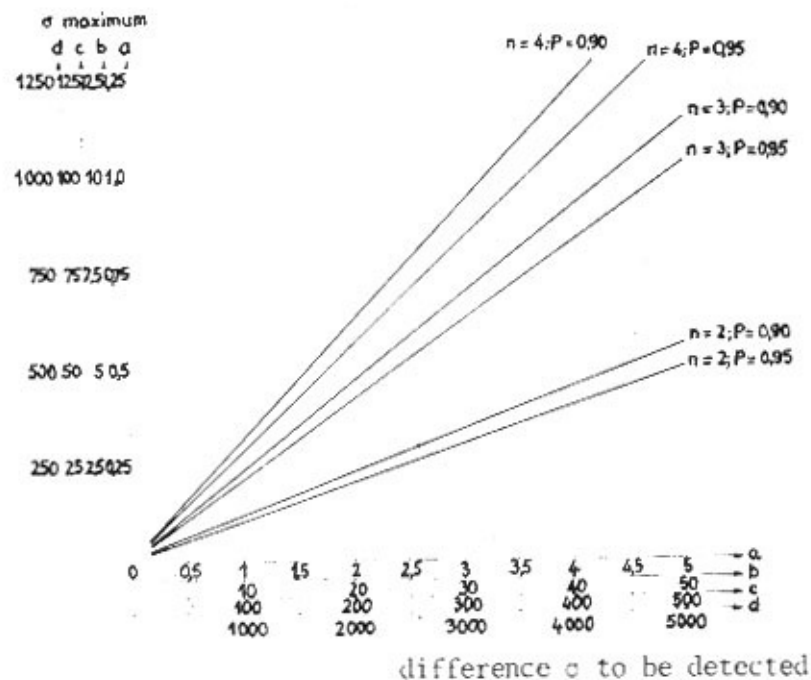
The term critical level is often used to indicate the concentration below which reductions in yield may be expected as a consequence of nutrient deficiency. BRAUD (8) defines the critical level as the content below which the yield is less than 90 % of that obtained with complete fertilization. The "nutrition index" for a given element is then

$$I = \frac{X_o}{X_c} \cdot 100 \quad \text{where } X_o \text{ is the observed concentration}$$
$$X_c \text{ is the critical level}$$

At this moment several valuable publications are available which contain a review of reference data for many crops and the mineral element contents of their tissues in varying nutrient situations. This represents a most useful collection of figures usable as references with regard to the analytical results obtained in plant testing services (7)(12)(13)(20). A review of such figures is given in Table 5 for several important crops and an exemple of more detailed information is included for coffee (Table 6). However transfer of interpretation tables may be dangerous due to year-to-year and location-to-location variations as a result of soil-climate-plant interactions.

In spite of this, TSERLING (37) stated that plants of the same species require the same amounts of nutrients for their normal growth and argued that their composition should be brought to one and the same optimum value irrespective of the soil on which they grow. Even in the most favourable conditions plant analysis can only give plant requirements, not soil requirements (BEAUFILS) (3) (4). Therefore plant tests can be very helpful in identifying an existing deficiency (or an excess), but when formulating fertilizer recommendations it is necessary to take also into account soil characteristics, experimental observation, variable experimental results and economical factors.

Table 5. Key data on nutrient element concentrations in plants

Crop	Element	Indicator part of plant	nutrient ranges %				References
			decifient	critical	normal optimal adequate	high	
Oil palm	N	Central leaflets of frond 17		<2.50	2.5-2.8		Chapman Okoye Prévot & Ollagnier Coulter Chemara de Geus
	P			<0.15	0.15-0.19		
	K'			<1.00	1-1.3		
	Ca			<0.60	0.6-0.70		
	Mg			<0.24	0.24-0.50		
Cocospalm	N	leaf n° 4 for palms up to 4 years old		1.70	1.8-2.0		Chapman de Geus Frémond & Nucé de Lamothe
	P			0.10	0.12-0.13		
	K	leaf n° 9 for 5 to 7 years old palms		0.45	0.8-1.0		
	Ca			<0.50	0.50		
	Mg			<0.35	0.35		
	Na	leaf n° 14 for older palms			0.40		
Rubber	N	leaves		<3.0	3-3.5	>3.5	Guha & Yeow Chapman de Geus RRIM Bolle-Jones
	P			<0.20	0.2-0.27	>0.27	
	K			<1.0	1-1.4	>1.4	
	Ca						
	Mg			<0.2	0.2-0.25	>0.25	
Olive	N	leaves		1.2	1.2-2.1		Bouat Prévot & Buchmann de Geus Chapman Samish et al
	P			<0.10	0.10-15		
	K			0.22-0.30	0.74-1.20		
	Ca				1.89-2.40		
	Mg				0.23-0.34		
Cacao	N	leaves	<1.80	1.8-2	>2		de Geus Murray
	P		<0.13	0.13-0.20	>0.20		
	K		<1.2	1.2-2	> 2		
	Ca		<0.3	0.3-0.4	>0.4		
	Mg		<0.2	0.2-0.45	>0.45		
Tea	N	first leaf with bud and third leaf		4.1	4.25-4.75		de Geus Willson Tolhurst
	P		<0.35	0.35	>0.35		
	K		<1.60	1.6-2.00	>2		
	Ca					0.3-0.4 Lin	
	Mg						

Crop	Element	Indicator part of plant	nutrient ranges %				References
			deficient	critical	normal optimal adequate	high	
Orange	N	4-7 old		<2.2	2.2-2.8	>2.8	Bergmann &
	P	leaves		<0.12	0.12-0.19	>0.19	Neubert
	K			<1	1-1.8	>1.8	Chapman
	Ca			<3	3-7	>7	
	Mg			<0.2	0.2-0.7	>0.7	
	S			<0.2	0.2-0.4	>0.4	
Banana	N	"zone 1/3"			2.6		Chapman
	P	one-third			0.18-0.2		de Geus
	K	section on		2.6-2.7	3.15-4.15		Osborne
	Ca	either side			2.02-3.4		Martin-Prével
	Mg	of the mi-drib			0.39-0.78		et al.
Pine apple	N	middle third section of white basal portion of the last fully developed leaf (D-leaf)		<1.6	1.6-2.2		de Geus
	P			<0.16	0.16-0.25		Chapman
	K		<3.4	3.4	3.5-4		Su
	Ca						Samuels &
	Mg				0.28-0.30		Gandia-Diaz
Sugar-cane	N	laminae of	<1	1-1.5	1.5-2	>2	Samuels
	P	leaves	<0.15	0.15-0.18	0.18-0.25	>0.25	de Geus
	K	3,4,5 and 6	<1.5	1.5-1.65	1.65-2	>2	Chapman
	Ca						
	Mg						

Table 6. Review of nutrient element contents in coffee

Element	Indicator part of plant	Nutrient ranges %				Reference
		deficient	critical	optimal	high	
N	fourth leaf	<2	2-2.3	2.3-2.8	>2.80	Chaverri et al
	"	-	2-2.5	2.6-3.0	>3.00	Malavolta
	"	<2	2-2.5	2.5-3.0	>3.00	Malachado
	"			2.0-3.25	>3.25	Cooil
	"	<2.2	2.2-2.6	2.6-3.4	>3.4	Southern
	youngest fully mature leaf		3.0			Espinosa
	fourth leaf		2.6	2.9-3.0		Culot et al Colonna
	third leaf		1.7			Borget
	leaves			2.70		Louê
	third leaf	1.5-1.8	1.8-2.5	2.5-3.0	>3.0	Müller
	eight pair from end of branch	1.65		2.70		Medcalf et al
	third pair		3.0	3-3.4		
P	fourth leaf	<0.09	0.09-0.12	0.12-0.2	>0.2	Chaverri et al
	"	-	0.05-0.10	0.11-0.15	>0.15	Malavolta
	"	-	0.11	0.11-0.15	>0.15	Malachoda
	"			0.08-0.15		Cooil
	"	<0.10	0.10-0.13	0.13-0.19	>0.19	Southern
	youngest fully mature leaf		0.12			Espinosa Culot et al
	fourth leaf	0.16	< 0.20			Colonna
	third leaf		0.07			Borget
	leaves			0.13-0.15		Louê
	third leaf	0.06-0.09	0.09-0.13	0.13-0.15	>0.15	Müller
	eight pair from end of branch	0.10		0.18		Jones
	first pair			0.11-0.12		
K	fourth leaf	<1	1-1.7	1.7-2.7	>2.7	Chaverri et al
	"	-	1.5-2.0	2.1-2.5	>2.5	Malavolta
	"	<1.1	1.1-1.5	1.5-1.8	>1.8	Machado
	"	<0.8	< 0.8	1-1.8	>2	Cooil
	"	<1.4	1.4-1.8	1.8-2.6	>2.6	Southern
	youngest fully mature leaf		2.3			Espinosa
	fourth leaf	<1.8	1.8	2.35		Culot et al
	third leaf		1.0			Colonna
	leaves			1.8-2.2		Borget et al

./..

Element	Indicator part of plant	Nutrient ranges %				References
		deficient	critical	optimal	high	
K	third leaf	<0.8	0.8-1.5	1.5-2.5	>2.5	Loué
	eight pair from end of branch			1.5		Müller
						Frankart &
	fourth pair			2.0		Croegaert
	third pair from apex	<1.0		1.8-2.3		Medcalf et al.

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IV. SETTING-UP SOIL AND PLANT TESTING SERVICES AT DIFFERENT LEVELS OF

SOPHISTICATION

Different levels of organisation of a soil and plant testing service can
be distinguished with regard to :
- type of analysis : restricted number of elements, simplified methods
- volume of activities : number of samples to be taken and analysed yearly
  and geographical area to be covered
- levels of sophistication : equipment, automation, data treatment, inter-
  pretation system etc.
The required instrumentation, housing and personnel must be estimated
consequently.

If it is decided to start a laboratory for a limited number of analysis
and samples per year, its expansion after a certain time of activity and
experience must be possible and therefore be conceived from the start.
Though the quality of its performances will progressively improve, the
accuracy of the work, the precision of the analysis and the reliability
of the advises must be of high quality, independently from size and orga-
nization level.

For this reason, even a starting service should not be too small. Its
size must be sufficient to make possible the acquisition and efficient
use of apparatus and full employment of a staff composed of personnel
with different specialization and technicity.

Another reason for considering a critical size is the financial aspect of
operation. It may certainly not be expected that a new laboratory could be
founded and repaid with the income of its activities. Even in the deve-
loped countries, such laboratories are established and supported either
by the government, by fertilizer industries or agricultural organizations.
However maintenance and creative activities will greatly be favoured if
they can be supported by some own earnings.

If it seems reasonable to distinguish between different levels of organi-
zation, the distinction should be on a quantitative as well as on a metho-
dological basis. An increased capacity will create a need to introduce

special devices for handling larger series. At the same time the problem of automation arises and it must be examined to which level of sophistication this can be extended.

The following paragraphs describe a standard concept quantitatively corresponding with a size and capacity of 10 000 samples yearly, as well as its possible extension to 30 000 samples.

In planning a laboratory it is efficient to work with modules. The modules used in figure 3 have 3 m x 4 m and each room has this size or a multiple of it.

1. General accommodation and furniture

The laboratory concept represented in fig. 3 has the following parts :

1.1. Section A :

- Rooms for sample reception, registration, pretreatment (soils and plants separately) and storage
- Workshop for mechanical reparations.

General equipment :

- 3 tables
- 8 racks (0,5 m depth) with minimum 5 levels at 30 cm distance for drying scales and for storage of sample boxes
- 1 cupboard for forms, labels, small equipment
- 1 blackboard in room A1 (against wall)
- 2 small carts for internal transport

1.2. Section B : Analytical work

- large room for chemical preparations
- 2 rooms for instrumental measurements
- rooms for water distillation (or de-ionization), wet destruction, storage of glassware, chemicals, spare parts
- Rooms B2, B3, B6, B7, B8 are separated by a glass and wood wall from B1.

General equipment :

- 4 completely equiped laboratory working tables (4 m x 1,5 m) (B1)
- appropriate racks in rooms B4 and B5
- working tables against the wall in rooms B2, B6 and B8
- 4 desks + 4 chairs in B2, B6, B7, B8
- 2 fix cupboards against walls (1) and (2) (0,5 m x 4 m) over full height
- 10 laboratory seats of 60 cm height
- 3 fix succion and vapour evacuation modules of 0,75 m.x 1,5 m (B1)
- succion systems mounted above flame photometer (B8), atomic absorption
  spectrometer (B8), muffle furnace and Kjeldahl rack (B10)
- 2 washing-sinks in room B9 for cleaning glassware.

1.3. Section C : offices

- library and documentation room, meeting and conference room (C4, C5)
- 1 room for head of the service (C3)
- 1 room for secretary and 1 for calculation and data-treatment (C1 & C2)

Remark : rooms C4 and C5 are separated by a movable wall.

General equipment

- 2 desks + 6 chairs (C2, C3)
- 2 typewriters desks + 2 chairs (C1)
- 2 book-cases + 2 document-racks
- 8 tables of (0,5 x 1 m) + 16 chairs (C4 and C5)
- 1 blackboard against wall in room C5
- 1 projector for 5 x 5 cm slides + screen (C5)
- 2 typewriters (C1)
- 2 calculating machines (C1 and C2)
- 1 copying machine
- 1 drawing desk
- facilities for storing standard forms and documentation
- if wanted fix cupboards may be placed in the main corridor (against
  rooms B4, B5, B6, B7)

1.4. Section D :

- cloakroom, toilets, shower with approrpiate equipment (D1 to D4)
- restroom : table + chairs for taking a simple meal (D5).

2. Internal laboratory organization

The operation of the soil and plant testing service comprises collection
of samples, analytical operations, treatment of results and finally inter-
pretation and fertilizer recommendation.
Each of these tasks needs organization, instrumentation and personnel in
function of the volume and required speed.

At the F.A.O.-expert consultation on soil and plant testing in developing
countries, held in Rome from 13 to 17 June 1977, a certain number of prac-
tical and organizational problems were raised. These problems concern the
accuracy of equipment and laboratory facilities, the maintenance of appa-
ratus, the availability of materials, spare parts and chemicals, the ade-
quate supply of water and electricity. The need of trained personnel, as
well laboratory techniciens as instrument mechanics, was also considered
to be a serious problem.

2.1. Choice of instrumentation for standard laboratory

Having decided which type of analysis must be carried out, the equipment
can be devided as follows :
- glassware and mechanical aids
- electro-mechanical apparatus : grinding mills, shakers, heating plates,
  waterbath, drying stove, electrical oven, etc.
- electro-chemical apparatus : potentiometer, pH-meter, conductivity meter
- spectrometric apparatus : colorimeter, spectrophotometer, flame photo-
  meter, atomic absorption spectrometer.

Each of these apparatus must be accompanied by a number of accessories,
without which they can not be used. It is as important to have sufficient
connecting wires, appropriate plugs etc. and this is different from country
to country.

In the actual conditions it is necessary to start with a basic equipment
and instrumentation which represents already a certain level of sophistica-
tion. Thus, flame photometry and atomic absorption are so much better
suited for the determination of essential elements such as K, Ca, Mg, Zn,
Cu and other trace elements than any other method, that these techniques
should not be withhold from the standard soil and plant testing laboratory.

In this sence a critical minimum is required for an efficient activity. A review of such equipment is given below, respectively for handling $\pm$ 10 000 and the necessary complement for $\pm$ 30 000 samples is also added. Taking into account unequal arrival, periods of variable activity due to maintenance, cleaning, rest, seasonal peaks and occasional charges, it is necessary to be armed for a temporary higher capacity.

2.2. Different levels of instrumentation

2.2.1. Simplified methods

In some circumstances it may be necessary to apply colorimetric methods for some elements, rather than atomic absorption spectrometry. Such techniques are excellent alternatives in case of break-down or shortage. EVENHUIS and de WAARD (18) have described a method for the determination of 9 elements (in plants) by flame photometry (K, Na, Ca) and colorimetry (N, P, Mg, Fe, Al, Mn). The reactions are carried out in test tubes and the number of reagent additions are kept to a minimum. Such simplified analysis are performed in the laboratory and with the same care and discipline as any other laboratory operation. Field methods for rapid tests at the farm, using portable soil testing kits are not being considered here. The experience shows that the latter techniques easily lead to erroneous results, unless executed by skilled and very disciplined persons.

2.2.2. Further sophistication

The question of automization and sophistication arises inevitably as soon as labour saving is considered. The latter is possible by simple mechanical and electrical apparatus as well as by heavy and more complicated instrumentation.

It should be kept in mind that the more instruments are complicated, the more they necessitate rigidly controlled working conditions e.g. environmental temperature and humidity, stabilized electrical sources and the more they are vulnerable. The following citations from soil scientists with sufficient practical experience in developing countries are eloquent :

- "In many laboratories, one can see expensive pieces of machinery lying idle, due to some minor defects. Getting a pH meter or spectrophotometer repaired becomes such a costly and time-consuming affair, that is is often easier to order a new one. The purchase of complicated instruments

in developing countries is to be discouraged ----- what the developing
countries need are simple instruments which can be operated and main-
tained easily . . . . ."   (R.G. MENON, 1977)(30).

- "Even in the absence of sophisticated instruments, it is possible to de-
velop systems which allow a large output with manual methods, perhaps
aided by "automation" to the extend of automatic dispensers and diluters
which are operated by hand" (G.W. ARNOTT, 1977)(2).

Many simple devices also contribute effectively to time and labour saving :
multiple racks for flasks for simultaneous filtration and further simul-
taneous handling.

Further automation and sophistication should only be introduced progressi-
vely and not without making sure that maintenance, repair and availability
of spare-parts are warranted. Recording of results is recommended as one
of the first steps in instrumental extension.
If the former conditions are definitely fulfilled and when sufficient expe-
rience has been accumulated in the local working conditions, apparatus such
as automatic colorimeters and automatic sample changers should be
taken into consideration.   Moreover it is necessary to stress that auto-
analyzer systems generally will need an important adaptation of the analy-
tical methods themselves. The further evolution of auto-analysis will be
largely dependent upon the commercial availability and local servicing fa-
cilities.

2.3. Suggested equipment for different capacities

10.000 samples yearly	supplement for 30.000 samples yearly
1. Glassware & mechanical aids	
test tubes : 2000 tubes of 15 ml	x 2,5
200 tubes of 25 ml	-
beakers : 250 beakers of 100 ml	
20 beakers of 500 ml	
20 beakers of 1 l	x 2
20 beakers of 2 l	
erlenmeyers (conical flasks) :	
250 erlenm. of 300 ml	
100 erlenm. of 150 ml	x 2
50 erlenm. of 500 ml	
volumetric flasks :	
200 flasks of 100 ml	x 2
200 flasks of 50 ml	x 2
20 flasks of 250 ml	x 2
20 flasks of 500 ml	x 2
20 flasks of 1000 ml	x 2
10 flasks of 2000 ml	x 2
5 flasks of 5000 ml	x 2
burettes 10 burettes of 25 ml	-
10 burettes of 50 ml	-
5 automatic burets of 50 ml	-
pipettes : 10 pipettes of 0,5 - 1 - 2 - 3 - 4 - 5 - 6 - 7 - 8 - 9 - 10 - 15 - 25 - 50 and 100 ml	x 2

10.000 samples yearly	supplementary for 30.000 samples yearly
bottles :	
1000 plastic bottles of 125 ml	x 2
100 plastic bottles of 250 ml	x 2
2 plastic water containers of 300 l with tap	x 2
10 plastic water containers of 10 l, with tap	x 2
25 plastic washing bottles of 500 ml	x 2
funnels :	
500 plastic funnels of Ø 10 cm	x 2
200 glass funnels of Ø 6 cm	x 2
5 excicators (capacity of 20 beakers of 100 ml)	x 2
4 USA-standard sieves of 2 mm and 0,5 mm	x 2
2 analytical balances, mounted on stabilized tables	+ 1
2 top pan precision balances on sturdy tables	+ 1
1 ion exchange unit for water purification of capacity 3000 l between 2 regenations	capacity of 6000 l per regeneration
filterpaper :	
for soil analysis :	
20.000 filters Whatman n° 40 or Schleicher & Schull n° 589/2 Ø 18 cm	x 2
for plant analysis :	
5.000 filters Whatman n° 42 or Schleicher & Schull n° 589/3 Ø 12 cm	x 2
Mortar with pestle	
Sieve	
Cardboard boxes (15 x 10 x 6 m) for storing soil samples	

10.000 samples yearly	supplementary for 30.000 samples yearly
2. Electro-mechanical apparatus	
1 vacuum cleaner	
1 drying stove of 200 l	+ 1
1 large drying stove for plant samples	
1 muffle furnace	+ 1
1 grinding mill	
2 programmed shakers for 24 conical flasks of 300 ml	+ 1
2 electrical hot plates	
1 electrical water bath	+ 1
1 plant tissue grinder	
6 magnetic stirrers	
1 centrifuge + centrifuge tubes of 50 ml, 100 ml and 250 ml	
1 vacuum pump (for extracting saturation soil extracts) and 1 filter funnel stand and funnels	
1 Kjeldahl apparatus (semi-automatic Kjeltec from Tecator or automatic Kjelfoss)	
10 dispensers (0 - 10 ml) manuel	+ 5
2 automatic dispensers	+ 2
2 automatic diluters	+ 2
2 variable proportion dose apparatus to prepare constant ratio soil extracts	+ 1
1 electronic calculator	+ 1

10.000 samples yearly	supplementary for 30.000 samples yearly

3. Electro-chemical apparatus

2 pH-meters, possibly with digital reading, fitted also for specific electrodes	+ 1
1 conductivity meter + 2 measuring cells	+ 1
electrodes for pH meter :	
4 reference electrodes, Sat. Hg_2Cl_2	+ 2
4 glass electrodes	+ 2
2 combined electrodes pH range 0-14	+ 1
specific electrodes :	
2 nitrate electrodes + reference electrodes ($HgSO_4$) or double junction	–
1 fluoride electrode	

4. Spectrometric apparatus

1 photo-colorimeter + cuvettes and autocell accessory	1 spectrophotometer + autosample or sample changer
1 flame photometer with air compressor, propane and acetylene	+ sample changer
1 atomic absorption spectrometer (+ hollow cathode lamps for Ca, Mg, Zn, Cu, Mn, Fe) with compressor, bottles of acetylene & gas-holder	2 atomic absorption spectrometers + autosampler and read-out system

2.4. Chemicals

2.4.1. Reagent grade (pro analysi)

Chemicals	for 10 000 samples	for 30 000 samples
H_2SO_4	10 l	20 l
H_3PO_4	10 l	20 l
HCl	5 l	10 l
HNO_3	10 l	20 l
NaOH	5 kg	10 kg
NH_4OH	5 l	10 l
$K_2Cr_2O_7$	6 kg	18 kg
DTPA	0.5 kg	1.5 kg
$K_4Fe(CN)_6.3H_2O$	250 g	250 g
$K_3Fe(CN)_6$	250 g	250 g
KH_2PO_4	250 g	250 g
KNO_3	250 g	250 g
Na_2SO_4	250 g	250 g
NaCl	250 g	250 g
NH_4Cl	250 g	250 g
$AgNO_3$	250 kg	250 g
Se	2 kg	6 kg
$FeSO_4.7H_2O$	1 kg	2 kg
$KMnO_4$	1 kg	1 kg
$ZnSO_4.7H_2O$	1 kg	1 kg
$CuSO_4.5H_2O$	1 kg	2 kg
$Co(NO_3)_2.6H_2O$	1 kg	1 kg
$Pb(NO_3)_2$	1 kg	1 kg
$NiSO_4.7H_2O$	1 kg	1 kg
$CdCl_2.2H_2O$	1 kg	1 kg
Phenolphtalein	250 g	500 g
methylred	250 g	250 g
bromocresolgreen	250 g	250 g
diphenylamine	250 g	250 g
EDTA (acid form)	3 kg	9 kg
Na_2EDTA	1 kg	3 kg

2.4.2. Purified chemicals (produits chimiques purs)

Product	for 10 000 samples	for 30 000 samples
$CaCl_2$	1 kg	2 kg
KCl	500 kg	1500 kg
H_2SO_4	450 l	1400 l
H_3PO_4	100 l	300 l
HCl	60 l	180 l
HNO_3	300 l	900 l
$Na_2S_2O_3$	50 kg	150 kg
H_3BO_3	12 kg	36 kg
NaOH	200 kg	600 kg
NH_4OH	400 l	1200 l
$Ca(OH)_2$	10 kg	30 kg
KCl	340 kg	1030 kg
K_2SO_4	100 kg	300 kg
$KAl(SO_4)_2.12H_2O$	15 kg	45 kg
$CaSO_4.2H_2O$	6 kg	18 kg
MgO (powder)	20 kg	60 kg
NH_4F	0.50 kg	1 kg
NH_4AC	80 kg	240 kg
$BaCl_2$	0.5 kg	1.5 kg
$NaHCO_3$	50 kg	150 kg
$(NH_4)_6Mo_7O_{24}.4H_2O$	1.5 kg	4.5 kg
$SnCl_2.2H_2O$	0.5 kg	1 kg
NH_4HCO_3	15 kg	50 kg
Devarda alloy	20 kg	60 kg
gelatine powder	0.5 kg	1 kg
buffer solution pH 4	1 l	3 l
activated charcoal	20 kg	60 kg
toluen	1 l	2 l
Mohr's salt $Fe(NH_4)_2(SO_4)_2$	40 kg	120 kg
salicylic acid	10 kg	30 kg
potassium biphtalate	1 kg	1 kg
ethanol 95 % (methylated spirit)	4000 l	12000 l
hydroxylamine hydro-chloride	0.5 kg	1.5 kg

salicylaldehyde	1 l	3 l
Acetic acid	10 l	30 l
thioglycollic acid	100 ml	500 ml
ascorbic acid	250 g	1 kg
4-amino-5-hydroxynaphtalin disulfonic acid : Merck Schuchardt (1973) n° 820078 AM 102	500 g	1 kg

Fig. 3. Laboratory lay-out
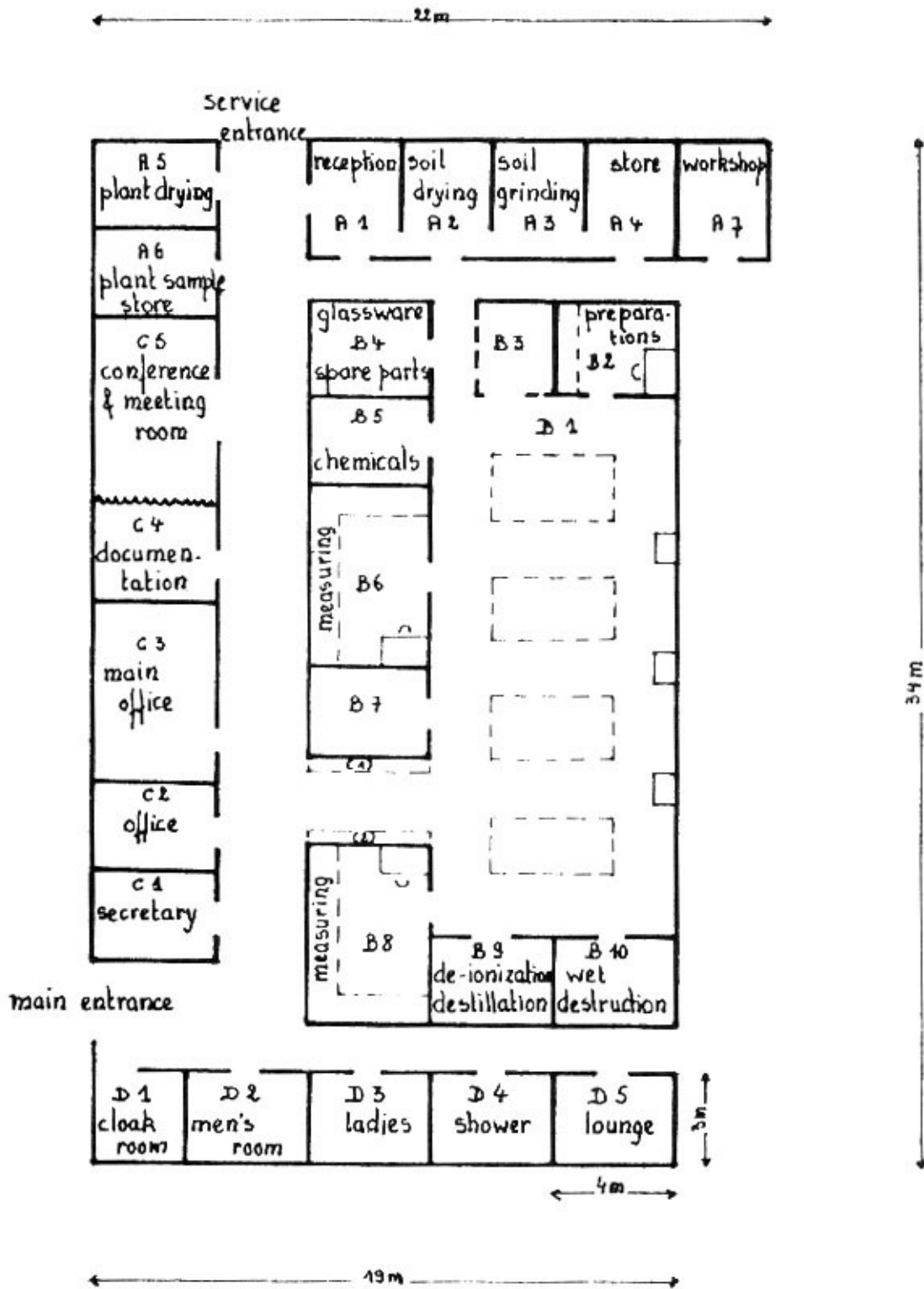
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PART II. ANALYTICAL METHODS

A. METHODS OF CHEMICAL SOIL ANALYSIS

1. Sampling and sample treatment

In order to obtain truly representative samples, they should preferably
be taken by a technician or assistant of the extension service, who is
trained to consider uniformity of the area, topography, texture, cropping
pattern etc.

Minimum 20 and preferably 40 carrots of soil from one parcel must be mixed
to make a representative sample (23,24)These carrots can be taken with
stainless steel tube samples (see model). The samples are packed in a special
sample bag and properly labeled. At the same time an information sheet is
completed (model A).

Drying : At their arrival in the laboratory soil samples are usually air-
        dried at a temperature between 25° to 35°, using plastic scales
        of 20 x 20 x 6 cm, fitting side by side in drying racks

Grinding : Soil aggregates are crumbled in a porcellan or agate mortar
          and passed through a 2 mm sieve.

Clay soils are best crushed for passing the sieve before they reach complete
-air-dryness, otherwise the crushing process is difficult.

The course fraction represents stones and gravel, the percentage of which
is eventually noticed.

The < 2 mm fraction is used for most of the analytical determinations
(pH, CEC, cations, soluble salts, etc.).

Homogenization of the fine soil for further analysis, especially of carbon
and nitrogen, is made with a mecanical grinding mill. If trace elements
are to be determined it is necessary to check the absence of contamination

at this stage. The grinding compartment must be ventilated and equiped
with a vacuum cleaner (industrial type).

After this first preparation the samples are put into labeled cardboard
boxes of 15 x 10 x 6 cm and stored. Sufficient storage capacity must be
available for the soil and plant samples of 1 year.

Equipment :

- Porcellan or agate mortar and pestle. Porcelain mortars are sometimes
  considered as possible sources of contamination for some soil analysis
  work
- 2 mm sieve
- soil mill.

2. Direct determinations

2.1. pH-values

When the soil sample is put in contact with water or with a neutral salt
solution, an equilibrium is reached, where the H^+-ions are distributed
between the liquid and solid phases.

With neutral salt solutions such as 1 m KCl or 0.01 m $CaCl_2$ more H^+-ions
are exchanged into the free solution.

Due to the action of the displacing ions K^+ and Ca^{++}, equilibrium is sooner reached in the latter case than with pure water.

The values called pH-KCl and pH-CaCl$_2$ are normally lower than the pH-H$_2$O. They are also more constant because the effect of small fluctuations is leveled off by the larger amounts of H^+ being present in solution.

The measurement in 0.01 m CaCl$_2$ was proposed because this corresponds with an equilibrium solution having a Ca^{++} concentration, the order of magnitude of which is comparable to a real soil solution.

It is easily understood that the measured values are normally in the following order : pH-H$_2$O > pH-CaCl$_2$ > pH-KCl

Measurements

pH-H$_2$O : Place 10 g air-dry soil in a 100 ml beaker and add 50 ml distilled water. The suspension is stirred and the pH measured after 18 hours of equilibration.

pH-KCl : Place 10 g air-dry soil in a 100 ml beaker and add 25 ml 1 n KCl. The pH of the suspension is measured after 10 minutes.

pH-CaCl$_2$: Place 10 g air-dry soil in a 100 ml beaker and add 25 ml 0.01 m CaCl$_2$.
Equilibrate for 10 minutes.

In each case the pH of the suspension is measured potentiometrically with a glass electrode versus a calomel reference electrode.

Before starting a series of measurements the potentiometer is calibrated with a buffer solution of known pH.

Equipment and reagents

- pH meter with glass and calomel electrodes
- beakers of 100 ml
- magnetic stirrer
- saturated KCl solution ($\pm$ 40 g/ 100 ml)
- buffer solution of pH 4 : the standard pH 4 buffer for calibration is 0.05 m potassium biphthalate. A stock solution of 0.3 m is prepared by dissolving 61.2 g of KHC$_8$H$_4$O$_4$ in 1 litre of hot water. A dilution of 6 times results in a 0.05 m solution
- pure water (distilled or de-ionized)
- 1 n KCl solution : dissolve 74.6 g KCl in distilled water and dilute to 1 litre
- CaCl$_2$ solution 0.01 m : dissolve 1.11 g CaCl$_2$ in a 1 litre volumetric flask.

2.2. Carbonates

The soil sample is treated with a known excess of a strong acid, which re-
acts with carbonates in the following way

$$CaCO_3 + H_2SO_4 \longrightarrow CaSO_4 + CO_2 + H_2O$$
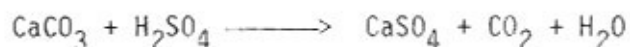
The excess H_2SO_4 is back titrated with NaOH

It is possible that some acid is also used in other reactions e.g. with cer-
tain minerals or for neutralization of eventually Na_2CO_3 in alkaline soils.

Procedure

Determination of carbonates
Place 1 g soil sample in a 300 ml
conical flask. Add 25 ml H_2SO_4 0.5 n
and bring the volume to ± 150 ml
with distilled H_2O. The erlenmeyer
flask is placed in a hot water bath
for 1 h. After cooling add 0.5 ml
mixed indicator and titrate with
0.5 n NaOH.
A blank (without soil) is run in the
same way as the sample.

Titration of 0.5 n NaOH solution
Pipette 100 ml 0.5 n HCl into 3 er-
lenmeyer flasks, add 0.5 ml mixed
indicator and titrate with 0.5 n NaOH.
When the red colour has turned to
green, the NaOH must be added drop-
wise. The endpoint is reached when
the colour changes to red.

Equipment

- 300 ml conical flasks
- hot water bath
- analytical balance sensitive to
  0.1 mg
- 100 ml pipette
- 50 ml burette for NaOH

- Reagents
  - H_2SO_4 0.5 n : dilute 13.9 ml con-
    centrated H_2SO_4 in 1 l distilled
    water and control by dilution with
    known base
  - NaOH 0.5 n : dissolve 20 g of NaOH
    in 1 l water and control with known
    acid
  - HCl 0.5 n
  - the indicator is prepared by mixing :
    . 100 ml phenolphtalein : 1 g in
      100 ml ethylalcohol (96 %)

. 60 ml methylred : 0.1 g in 100 ml
  ethylalcohol (96 %)
. 40 ml bromocresolgreen : 0.04 g
  bromocresolgreen + 80 ml H_2O +
  5.7 ml 0.01 n NaOH

Calculation :

If the normality of the NaOH-solution is t, while a ml were added to the
blank and b to the sample, the amount of H_2SO_4 which has reacted with the
soil carbonates is

$$(a - b). t \quad \text{milli-equiv}$$

Thus 1 g soil contains $(a - b).t$ milli-eq $CaCO_3$
or $(a - b) . t . 50$ mg $CaCO_3$

100 g soil contain $(a - b) .t . 5$ g $CaCO_3$ (%)

Remark : If less than 5 ml NaOH are needed for the titration, the determi-
nation should be repeated with 0.5 g soil.

.3. Soil organic matter (WALKLEY & BLACK)

Soil organic matter (humus) content is estimated from the determination of
carbon, which is made by oxidation under standardized conditions with po-
tassium dichromate in sulphuric acid medium.
The principle of this method is formulated as follows :

$$4 \ (Cr^{6+} + 3 \ e \longrightarrow Cr^{3+})$$
$$3 \ (C - 4 \ e \longrightarrow C^{4+})$$
$$\overline{4 \ Cr^{6+} + 3 \ C \longrightarrow 4 \ Cr^{3+} + 3 \ C^{4+}}$$

$$2 \ K_2Cr_2O_7 + 3 \ C + 8 \ H_2SO_4 \longrightarrow 2 \ K_2SO_4 + 2 \ Cr_2(SO_4)_3 + 3CO_2 + 8H_2O$$
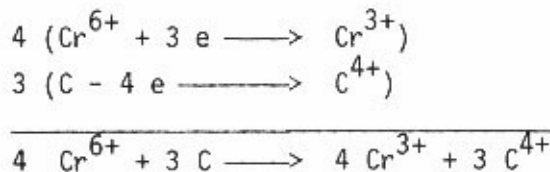
Normally 1 g air-dry soil is being used, but if the soil is poor or high
in organic carbon, more or less can be taken.

The soil sample is treated with a measured amount of $K_2Cr_2O_7$ in excess in the presence of H_2SO_4. H_3PO_4 is added in order to complex the Fe^{3+} ions which are liberated.

After 30 minutes, diphenylamine indicator is added and the excess $K_2Cr_2O_7$ titrated with a ferrous solution. As soon as ferrous ions are added in excess, the indicator turns from blue to a brilliant green colour.

$$Cr^{6+} + 3\ e \longrightarrow Cr^{3+}$$
$$3(Fe^{2+} - 1\ e \longrightarrow Fe^{3+})$$
$$\overline{Cr^{6+} + 3\ Fe^{2+} \longrightarrow Cr^{3+} + 3\ Fe^{3+}}$$

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$
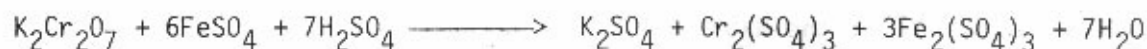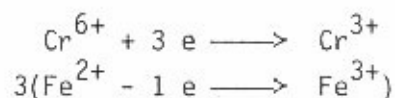
Procedure

Place 1 g or less (depending on the C-content of the soil) soil sample in a 500 ml erlenmeyer flask and add 10 ml 1 n $K_2Cr_2O_7$ solution. Add 20 ml concentrated H_2SO_4 and mix gently. The mixture is allowed to stand for 30 minutes on an asbest plate and then 150 ml H_2O and 10 ml concentrated H_3PO_4 is added. Add 1 ml diphenylamine indicator and titrate with 1 n Mohr's salt solution until a brilliant green colour is observed.

A blanc titration, without any soil, is carried out in the same way.

Equipment

- Analytical balance
- 500 ml erlenmeyer flasks
- pipette of 10 ml
- burette for the ferrous solution, preferably self-adjusting to zero
- magnetic stirrer with incorporated light
- Reagents :
  - 1 n $K_2Cr_2O_7$: dissolve exactly 49.04 g $K_2Cr_2O_7$ (previously dried at 200°C) in 1 l distilled water
  - concentrated H_2SO_4 (98 %)
  - H_3PO_4 85 %
  - diphenylamine indicator : dissolve 0.5 g diphenylamine in a mixture of 20 ml dist. water and 100 ml concentrated H_2SO_4
  - 1 n $Fe(NH_4)_2(SO_4)_2.6H_2O$ (Mohr's salt)[*] : 392 g Mohr's salt in 1 litre dist. water containing 20 ml H_2SO_4 18 N.

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[*] In case of unavailability of ferrous ammonium sulphate (Mohr's salt : $Fe(NH_4)_2(SO_4)_2.6H_2O$ it is also possible to use $FeSO_4.7H_2O$ (278 g $FeSO_4.7H_2O$ in 1 litre dist. water containing 5 ml concentrated H_2SO_4).

Calculations

If the $FeSO_4$-solution is t normal, a ml are used for the blank titration and b ml in presence of the soil sample.

The blank corresponds with exactly 10 milli-eq $K_2Cr_2O_7$, equivalent with the added amount of a.t.milli-eq Fe^{++} and the titration difference is (a.t - b.t) milli-eq.

For a.t milli-eq the titration difference is (a.t - b.t) milli-eq

$$10 \text{ milli-eq the titration difference is } \frac{10 \ (a.t - b.t)}{a.t} \text{ milli-eq}$$

$$\text{or } \frac{10 \ . \ (a - b)}{a} \text{ milli-eq}$$

Since 1 milli-eq $K_2Cr_2O_7$ = 1 milli-eq C = $\frac{12}{4}$ or 3 mg C the amount of oxidized C in 1 g soil is $\frac{10 \ . \ (a - b) \ . \ 3}{a}$ mg C or $\frac{(a - b)3}{a}$ % oxidized C

This calculation shows that the concentration of the ferrous-sulphate solution does not need to be precisely 1 n, which is a practical advantage. It should be kept in mind that the method of WALKLEY & BLACK is a conventional one. The oxidation procedure yields about 75 % of the total organic carbon present. This is usually taken into account by setting the carbon equivalent at 4 instead of the stoechiometric value 3. Thus the result is found as % C = $\frac{(a - b).4}{a}$. Assuming that oxidizable soil organic matter consists of 50 % C the humus content is found as % C x 2.

2.4. Total nitrogen

Determination of total nitrogen is rather intended to estimate the C/N ratio in the soil, than for determining the nitrogen requirement of soil and crops. In addition to total nitrogen, it may be useful to determine also the ammoniacal and nitrate forms, as described under n° 3
The method described here makes use of the classic Kjeldahl destruction of organic matter by oxidation with boiling sulphuric acid in the presence of a catalytic mixture, which raises the boiling temperature.
Two variants of this procedure are given to be used respectively if nitrate (and nitrite) may be neglected or is to be taken into account.

In the original Kjeldahl method nitrates and nitrites are transformed into the respective acids and evaporated. In the second (modified) procedure the digestion is carried out in presence of salicylic acid, which binds'-NO$_2$ from nitrates and nitrites as follows :

$$C_6H_4 \diagdown{}^{COOH}_{OH} \quad + H_2SO_4 \quad \longrightarrow \quad H_2O + C_6H_3 \diagdown{}^{COOH}_{OH}_{HSO_3}$$

$$C_6H_3 \diagdown{}^{COOH}_{OH}_{HSO_3} \quad + HNO_3 \quad \longrightarrow \quad H_2O + C_6H_2 \diagdown{}^{COOH}_{OH}_{HSO_3}_{NO_2}$$
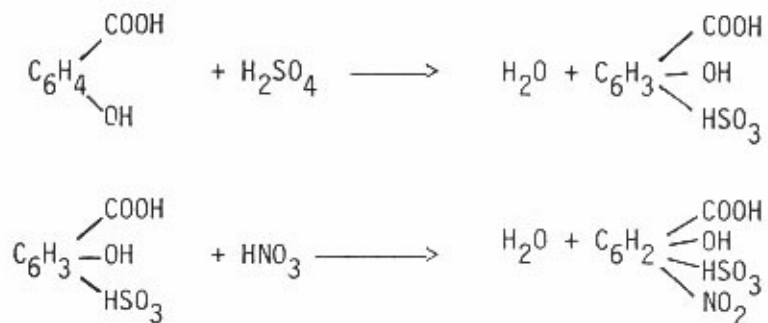
This complex is afterwards treated with Na$_2$S$_2$O$_3$, reducing -NO$_2$ into -NH$_2$, which is also forming (NH$_4$)$_2$SO$_4$.

The digestion may be carried out in Kjeldahl flasks of 200 ml, though it is recommended to use rather the modern device with 250 ml cylindrical tubes of special glass, fitting in an electrical destruction blok as shown in fig.

After destruction the mixture is cooled till room temperature, made alcaline with NaOH- and the flask is immediately connected with the distillation apparatus.

$$(NH_4)_2SO_4 + 2 \ NaOH \longrightarrow Na_2SO_4 + NH_3 + H_2O$$
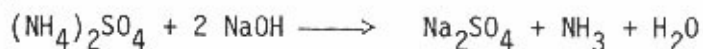
The released ammonia is quantitatively captured in an excess of boric acid in presence of a mixed indicator and finally titrated with standard acid (0.01 n HCl). The latter reacts with the formed ammoniumborate, which is reconverted into H$_3$BO$_3$ at pH 5.

Ammonia capture :

$$H_3BO_3 + NH_3 \longrightarrow NH_4H_2BO_3$$

Titration with HCl

$$NH_4H_2BO_3 + H^+ + Cl^- \longrightarrow H_3BO_3 + NH_4^+ + Cl^-$$
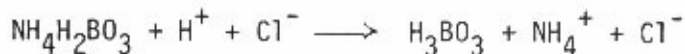
Procedure

Exactly 2 g air-dry soil (<1 mm) are treated in a 200 ml Kjeldahl digestion flask with 20ml sulfuric acid-salicylic acid mixture. After 30 minutes, add 5 g $Na_2S_2O_3$ and shake. After another 15 minutes, add 10 g K_2SO_4 and 0.1 g Se as catalysts. or mixture Merck n° 8030.
Heat the flask on a digestion rack until the solution turns clear.
After cooling add carefully 30 ml H_2O, alkalize the solution with 60 ml 30 % NaOH and start the steam distillation immediately, taking care that the glass receiver tube is immersed into the collecting solution. Collect the distillate into a 250 ml erlenmeyer flask containing 10 ml boric acid indicator mixture. After distillation of all NH_3, titrate the boric acid solution with 0.01 n HCl. At the endpoint the indicator turns from green to red.

Equipment

- analytical balance
- Kjeldahl digestion rack
- steamdistillation apparatus
- 200 ml Kjeldahl digestion flasks
- 250 ml erlenmeyer flasks
- burette and pipette

The modern device is a further development of the conventional Kjeldahl method. The digestion takes place in an electrical destruction bloc. The distillation is a distilling unit , a system in which sodium hydroxide is automatically dispensed and steam automatically generated into the sample solution.

- Reagents :

  - sulfuric acid-salycilic acid mixture : 50 g salic. acid in 1 l H_2SO_4 (d= 1.84)
  - K_2SO_4 and selenium or Merck mixture n° 8030
  - 30 % NaOH
  - indicator : prepare a mixture of equal volumes of methylred (0.66 °/°°) and bromocresolgreen (0.99 °/°°) in 95 % ethylalcohol
  - boric acid - indicator mixture : 20 g H_3BO_3, dissolved in 600 ml distilled water, are mixed with 10 ml indicator and diluted to 1 l with distilled water
  - 0.01 n HCl
  - $Na_2S_2O_3$

Choice of apparatus :

- Digestion system DS 20 Tecator
- Distilling Unit Kjeltec System II Tecator

Calculations :

If a ml 0.01 n HCl were used for titration the amount N is found as

$$a \times 0.01 \text{ milli-eq}$$
$$\text{or} \quad a \times 0.01 \times 14 \text{ milligr. N}$$

Working with p g soil the N content is

$$\frac{a \times 14}{p} \text{ mg N per 100 g soil}$$

2.5. Cation exchange capacity (C.E.C)

There are a multitude of methods available for determination of the cation exchange capacity of soils and this publication doesn't intend to review them even partly.
The general principle consists in saturating initially the adsorption complex with one single ion and determining the latter after quantitative removal by another displacing ion. Ammonium and barium are often used as displacing ions.

In practice there are three different approaches to the problem :

a) summation methods : the exchangeable cations can be displaced with a saturating salt, and the CEC is taken as equivalent to the sum of exchangeable cations in the extract.

b) displacement of the index cation after washing out excess salt : when the exchange sites have been saturated with an index cation, the soil is washed free of excess saturating salt and the amount of index cation adsorbed by the soil can then be displaced and determined. There are a number of variations, of which two are of particular interest :
- the NH_4OAc method, probably the most widely used
- the $BaCl_2$ - triethanolamine method.
In the NH_4OAc method the excess saturating salt is removed with 95 % ethanol. Water may not be used   due to the hydrolysis reaction :
Soil NH_4 + H_2O $\rightleftharpoons$ Soil-H + NH_4OH
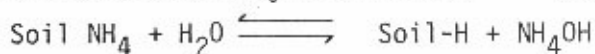
c) radioactive tracer method : the most accurate method, but due to its complicated and expensive procedure, more often used for research than for routine analysis.   *
                              *   *

The most widely used CEC method for soils is the NH_4OAc method. Briefly the principle consists in saturating the adsorption complex with NH_4^+ ion, washing the excess NH_4^+ with ethanol, and determining the adsorbed NH_4^+ ion after quantitative removal by K^+ ions.

Procedure

Weigh 10 g air-dry soil (<2 mm) into a beaker and mix with 25 g quartz sand previously washed with HNO_3 and distilled water. Place the mixture quartz-soil quantitatively into a prepared percolation tube. Use a sintered glass-disc or glasswool as a support for the soil column. Finally place 10 g of quartz on top of the mixture.

Percolate the soil with 250 ml 1 N NH_4OAc pH = 7. For calcareous soils (>5 % $CaCO_3$) use 500 ml ammoniumacetate solution. The percolates may be used for determination of exchangeable cations in common soils. Due to dissolution of carbonates this method is not suited for determina-

Equipment

- percolation tubes of 20 mm diameter and 450 mm height.
- glass-wool or sintered glass discs
- analytical balance
- 500 ml volumetric flasks
- 500 ml erlenmeyer flasks
- steam distillation apparatus
- burette, pipettes.

Reagents

- 1 N NH_4OAc pH = 7 : prepare a sufficient volume by mixing 70 ml NH_4OH, specific gravity 0.90, and 58 ml 99.5 % acetic acid per litre of solution desired. After cooling, adjust exactly to pH = 7 and dilute to volume with water
- quartz sand

tion of exchangeable cations in calcareous soils

The rate of percolation should be as constant as possible for all soils. Therefore it may be necessary to mix more quartz with the soil in the case of heavy soils. The rate may also be kept constant by means of a stopcock placed at the outlet of the percolation tube

After treatment with 1 n NH_4Ac pH=7 the adsorption complex is saturated with NH_4^+ ions. The excess of soluble ions is removed by washing with 400 ml ethanol 95 % added in fractions of 30-40 ml. Discard the filtrate.

The NH_4^+ saturated soil is then treated with 500 ml 1 N KCl in order to displace adsorbed NH_4^+ ions. Collect the percolate into a 500 ml volumetric flask. After percolation make up the final volume till the mark with 1 N KCl solution.

Determine NH_4^+ in the percolate by distillation. Therefore pipette 10 ml of the percolate into a distillation flask ; add 2 drops of phenolphtalein and 1 g MgO powder and distill immediately. Collect the distillate in an erlenmeyer containing 10 ml 3 % boric acid indicator mixture and finally titrate the borate solution with 0.01n HCl.

Depending on the NH_4^+ content, the volume of percolate to be distilled may vary.

- 95 % ethylalcohol
- 1 N KCl solution = 74.55 g/litre
- Reagents and apparatus for NH_4^+ determination
- MgO powder
- Phenolphtalein indicator : a 0.1 % solution in 70 % ethanol (0.1 g in 100 ml)

- Indicator : a mixture of equal volumes of methylred 0,66 °/°° and bromocresolgreen (0,99 °/°°) in ethanol (95 %)
- Boric acid 2 % in distilled water containing 10 ml indicator per litre
- 0.01 n HCl

Calculations :

If the determination is made with 10 g soil and the final volume is 500 ml
1 n KCl, 10 ml of the latter solution correspond with $\frac{10}{50}$ g soil.
These $\frac{10}{50}$ g soil retain V . 0.01 meq NH_4^+ (V = ml HCl titrated)
In 1 g soil there are $\frac{V \cdot 0.01}{10/50}$ meq NH_4^+

100 g soil correspond with $\frac{V \cdot 0.01 \cdot 100}{10/50}$ or 5 . V milli-equivalents.

The CEC is 5 V milli-eq per 100 g soil.

Remark :

NH_4^+ in the final percolate can also be determined by means of the less
time consuming potentiometric procedure, using the specific ammoniak elec-
trode. Since in the final step of the CEC procedure a 1 n KCl solution is
used for the displacement of adsorbed NH_4^+, the potentiometric determina-
tion of NH_4^+ may be carried out in the same way as described (see 3.3).

2.6. Exchange acidity and lime requirement

The difference between the cation exchange capacity (T) of a soil and the
sum of exchangeable bases (S) is called exchange acidity and this is of
course also expressed in milli-equivalents per 100 g soil.

$$\text{Exchange acidity} = (T - S) \text{ milli-eq/100 g}$$

Both hydrogen and aluminium ions contribute to its value, which is related
to the difference between pH-H_2O and pH-KCl, though this difference does
not permit to calculate the exchange acidity.
Satisfying laboratory methods for the determination of lime requirement
were worked out since many years and they are still of general application.
Two techniques are described here :

2.6.1. Calculation of lime requirement from exchange acidity determination

The exchange acidity is determined by titration with NaOH after displace-
ment with 1 n KCl solution.

Procedure	Equipment and reagents
100 g soil sample are treated in a 500 ml flask with 250 ml 1 n KCl and shaken for 1 hour. After filtration through a dry plied filter into a dry erlenmeyer, 125 ml of the filtrate are titrated with 0.1 n NaOH in presence of phenolphtalein indicator (y ml)	- flasks of 500 ml - shaking apparatus - filter paper Schleicher & Schüll n° 597 1/2 Ø 185 mm - erlenmeyers of 300 ml - 1 n KCl : 74.5 KCl per liter - 0.1 n NaOH - phenolphtalein indicator : 0.1 % in 70 % alcohol.

Calculation :

- 1 ml 0.1 n NaOH (0.1 milli-eq) corresponds with 5 mg $CaCO_3$ or
  2.8 mg CaO.

- y ml 0.1 n NaOH were needed to neutralize 50 g soil (125 ml filtrate)

(a) 50 g soil need for neutralization $\quad$ y x 2,8 mg CaO

(b) 100 g soil $\quad$ " $\quad$ " $\quad$ " $\quad$ y x 5,6 mg CaO

Since 1 mg/100 g soil corresponds with 30 kg/ha : y x 168 kg CaO
are theoretically needed per Ha.

However, due to the fact that equilibration of the soil sample with
KCl, is not giving the total exchange acidity a conversion coefficient
of 3,5 from (a) to (b) is used instead of 2.

This gives a practical lime requirement of y x 294 kg CaO/ha.

$\qquad\qquad\qquad\qquad$ or y x 525 kg $CaCO_3$/ha.

2.6.2. Direct lime requirement determination

Portions of 10 g soil are equilibrated with increasing quantities of
$Ca(OH)_2$ in solution. After 3 times 24 hours the pH of the suspensions is
measured and the lime quantity for neutralization graphically determined.
The method is longer and therefore possibly less appropriate for serial
work.

Procedure	Equipment and reagents
Six 10 g portions of air-dry soil sample are treated in shaking flasks with respectively 0, 10, 20, 30, 40, 50 ml $Ca(OH)_2$ 0.04 n solution and the volume is brought to 50 ml with water. After 3 days equilibration with occasionally shaking, the pH of the suspensions is measured.	- Erlenmeyers of 150 ml - pH meter with glass and calomel electrode - shaking apparatus - 0.04 n $Ca(OH)_2$: saturated solution in CO_2 free water (theoretical content 1,52 g/l). Determine precise concentration by titration with standard 0.1 n HCl - 0.1 n HCl - buffer solution of pH 4.

Calculation :

$Ca(OH)_2$ concentration in milli-equiv. per liter = C (= ± 40)

ml $Ca(OH)_2$ solution added per 100 g soil	corresponding mg $CaCO_3$ added per 100 g soil	pH
0	0	
10	5 C	
20	10 C	
30	15 C	
40	20 C	
50	25 C	

Plot the pH values against the figures in column 2 and read the number of milligrams $CaCO_3$ required to bring the pH to the chosen level :

$$M \text{ mg } CaCO_3 \text{ per 100 g soil}$$

or $\quad 30.M \text{ kg } CaCO_3 \text{ per } 3.10^6 \text{ kg soil (1 Ha)}$

equivalent with $16,8 . M \text{ kg } CaO \text{ per } 3.10^6 \text{ kg soil (1 Ha)}$

This is a theoretical quantity which will not give in practice the same pH increase of the soil, due to the lower effectiveness of lime in field conditions. It is common to multiply the calculated requirement with a factor 2 for field dressings.

2.7. Gypsum requirement (23)

When a soil containing an excess of Na is shaken with a gypsum solution, Na is exchanged for Ca. The gypsum requirement of the soil is calculated in function of this exchange.

Procedure

5 g soil are treated in a 200 ml conical flask with 100 ml gypsum solution and shaken for 30 minutes.
Ca is determined in the original gypsum solution (A) and in the filtrate (B meq per 1).

Gypsum requirement =
2 (A - B) meq per 100 g soil

Equipment and reagents

- 200 ml conical flasks + stopper for extraction
- Gypsum solution : shake 5 g $CaSO_4.2H_2O$ in 1 l of water for 1 hour. (approximately saturated or minimum 28 meq per litre).

Remark :

The analysis can be done by flamephotometry, by atomic absorption spectrometry or by titration with complexon (versenate).

2.8. Conductivity and soluble salts

Specific electrical conductivity of a solution is directly related to its
ion content. It is measured with a conductivity cell and expressed in
millimhos or milliSiemens (mS) per cm (*)

$$1 \text{ Mho} = 1 \text{ Siemens} = \frac{1}{\text{Ohm}}$$

Due to the fact that conductivity measuring cells show individual deviations,
they must be calibrated with a 0.1 n KCl standard solution.
The conductivity of the soil solution gives an estimate of the total amount
of soluble salts. In practice measurements are made in saturation extracts
or in extracts obtained at soil/water ratios of 2/5 or 1/5. Saturation ex-
tracts give more reliable values, because the measurements are in better
correspondence with real field conditions.

Procedure

Introduce 200 g soil in a cylindri-
cal vessel of 12 cm diameter and 8
cm height. Add slowly distilled water
by means of a burette untill comple-
te saturation of the soil paste,
which then is wet enough to glisten,
flows slowly together when shaken
and slides off the spatula.
Note the volume of added water and
cover the scale. Allow the soil paste
to equilibrate for 1 night. The paste
is then filtered through a Büchner
funnel by suction and the filtrate
collected.
The conductivity meter is calibrated
with a solution of 0.1 n KCl (see

Equipment and reagents

- cylindrical vessels of 12 cm dia-
  meter and 6 to 8 cm height.
- burette of 100 ml
- vacuum pump
- Buchner funnels of 10,5 cm Ø and
  flasks of 150 ml
  (Buchner filter funnel stand)
- Filter papers to fit the funnels
  (9 cm diameter SS 589[3])
- Conductivity meter
- Conductivity cell
- Thermometer (room temperature)
- 0.1 n KCl : dissolve 7,455 g KCl
  in distilled water and dilute to 1 l

(*) Indication on scale of conductivity apparatus is either in (milli)mho
or in (milli)Siemens according to its origin.

table). The conductivity of the ex-
tract is measured, taking care that
the cell is completely filled.

Remarks   :

a) In order to correct the readings obtained with the conductivity cell the
latter is calibrated with a 0.1 n KCl solution at known temperature. The
specific conductivity of 0.1 n KCl is

temp (°C)	mmho/cm (mS)		temp (°C)	mmho/cm (mS)
5	8.22		20	11.67
10	9.33		21	11.91
15	10.48		22	12.15
16	10.72		23	12.39
17	10.95		24	12.64
18	11.19		25	12.88
19	11.43		26	13.13

If the check scale reading is S (24°C) the correction coefficient to be
applied for all further measurements is $\frac{12,64}{S}$

b) If the measurements are made in a 1/5 or 2/5 soil/water extract,
10 or 20 g soil are weighed and transferred into an appropriate jar.
After 30 minutes shaking the mixture is allowed to stand overnight. Then
the liquid phase is carefully separated by decantation into a cylindrical
vessel and the conductivity measured (+ temperature).

2.9. Redox potential

Meaning and principles

The redox potential of a soil is related to its aeration and represents a quantitative estimation of its state of reduction.

It is an intensity factor, because it reflects an actual and temporary situation, which can change when hydric and aeration conditions are varying.

Redox potential may be an important parameter in the characterization of paddy-soils or soils with poor oxygen diffusion in general.

A reduced soil is grey or greenish-blue, has a low redox potential and contains reduced counterparts of NO_3, Mn^{4+}, Fe^{3+}, SO_4 and CO_2 such as NH_4^+, Mn^{2+}, Fe^{2+}, S^{2-} and CH_4.

For the reduction $\quad Ox + ne \rightleftharpoons Red$

The redoxpotential Eh can be given as

$$Eh = Eo + \frac{RT}{nF} \ln \frac{(Ox)}{(Red)}$$

in which : Eh is the potential measured with a platinum electrode against the standard hydrogen electrode; (Ox) and (Red) are the activities of the oxidized and reduced species, Eo is the standardpotential measured when (Ox) and (Red) are equal, R the universal gas-constant and F is the Faraday constant. Eh is a quantitative measure of the tendency of a given system to oxidize or reduce susceptible substances.

Eh is positive and high in strongly oxidizing systems ; it is negative and low in strongly reducing systems. There is however no neutral point as in pH. Any chemical reaction involving the exchange of electrons will be influenced by redox potential (Eh).

The redox potential is measured with an electrode pair consisting of an inert electrode and a reference electrode (usually the saturated calomel electrode) by means of a high impedance potentiometer such as a pH meter.

The inert electrodes used commonly are bright platinum or gold.

In practice intrinsic and extrinsic errors deprive Eh measurements in most natural media of precise thermodynamic significance. Intrinsic errors include electrode malfunctioning (although Pt or Au-electrodes are truly inert, in contact with some ions, however, they may become coated by sulfides or chloride precipitates), pH effects, absence of true equilibrium, liquid junction potential errors and heterogeneity of the medium.

Procedure

1. directly in soils or sediments

Introduce a platinum electrode and a reference (SCE) electrode into the soil layer to be measured. Connect the leads to a suitable portable battery operated potentiometer and record the potential after stabilization

2. in soil or sediment samples

As already mentioned sampling is the most initial manipulation when redox-potential has to be measured.
All precautions should be taken to avoid contamination by air oxygen. Samples can be stored under nitrogen atmosphere and special electrode set ups are available to measure redox potentials in closed systems.

Remark

To check the apparatus its is advised to measure the redox potential of the redox standard solution.

Equipment and reagents

- portable battery operated poten- tiometer (pH-meter) with expanded millivolt scale
- platinum electrode
- saturated calomel electrode

Redox standard solution :
0.0033 m salts in 0.1 m KCl.
Dissolve exactly 1.394 g $K_4Fe(CN)_6 \cdot 3H_2O$, 1.087 g $K_3Fe(CN)_6$ and 7.455 g

A solution of 0.0033 m $K_3Fe(CN)_6$ and | KCl in distilled water and dilute to
0.0033 m $K_4Fe(CN)_6$ in 0.1 M KCl has an | 1 litre.
Eh of 0.430 V at 25°C.

Expression of results :

In most cases the redox potential is measured against a saturated calomel
electrode (SCE), this potential Ec is related to the redox potential Eh
(measured against a standard hydrogen electrode) through the following
equation

$$Eh \ (mV) = Ec + 242$$

242 mV being the difference between the potential of a standard hydrogen
electrode and the SCE at 25°C.

3. Determination of nutrient elements based on extraction

3.1. Extraction and determination of phosphorus

3.1.1. Method of Olsen

Extraction

5 gram soil are suspended in 100 ml
extracting solution together with 1
teaspoon of carbon black. Shake the
suspension for 30 minutes and filter
through a Whatman n° 40 or other
suitable filter paper. It the fil-
trate is not clear, add some more
carbon black and filter again.

Determination : The following proce-
dure is generally used with Olsen's
soil extracts :
Pipette a 5 ml aliquot of the clear
filtrate into a 25 ml volumetric
flask, add slowly 5 ml NH_4-molybdate
solution. Shake gently and make up
the volume to about 22 ml with dis-
tilled water. Add 1 ml diluted $SnCl_2$
solution and make up the final volume
with distilled H_2O. Shake vigorously
and measure the absorbance at 660 nm
after 10 minutes.

Standard series :
Pipette 0 - 1 - 2 - 5 - 10 ml of the
diluted P solution(2 ppm) into 25 ml
volumetric flasks. Add 5 ml $NaHCO_3$ so-
lution, 5 ml NH_4 molybdate solution,
distilled water to a volume of $\pm$ 22 ml.
Add 1 ml diluted $SnCl_2$ solution and
finally distilled water till the mark.
Measure the absorbance.

Reagents

- 0.5 M $NaHCO_3$ (42 g per litre)solu-
  tion adjusted to pH 8.5 with NaOH
- activated charcoal, tested for
  absence of phosphate
- ammoniummolybdate solution
  $(NH_4)_6Mo_7O_{24}.4H_2O$: dissolve 15 g
  in 300 ml hot distilled water. Af-
  ter filtration, add 342 ml concen-
  trated HCl to the cold solution and
  make up the final volume to 1 litre.
- $SnCl_2.2H_2O$: concentrated solution:
  dissolve 10 g in 25 ml concentrated
  HCl. Store in the refrigerator.
- $SnCl_2$ diluted solution : add 0.5 ml
  concentrated solution to 66 ml dis-
  tilled H_2O. This solution should be
  freshly prepared for each series
  of determinations.
- Standard P solution (100 ppm P) :
  dissolve 0.4393 g KH_2PO_4 in 1 litre
  distilled H_2O. Add a few drops of
  toluene.
- Diluted P solution (2 ppm) : 20 ml
  standard solution/1 litre H_2O.

Apparatus :
- 250 ml extraction bottles
- end-over-end shaker
- filter funnels (∅ 7 cm) and Whatman
  n° 4 filter paper (∅ 11cm)
- 25 ml volumetric flasks
- pipettes

3.1.2. Method of Bray und Kurtz

Extraction

Weigh 2 g of air-dry soil passed through a 2 mm sieve into a 50 ml conical flask and add 20 ml of the extracting solution, stopper the bottle and shake for 1 minute. Filter through a dry Whatman n° 42 filter paper. The filtrate should be clear. If not the solution is quickly poured back through the same filter.

Determination

Pipette a 1 ml aliquot of the clear filtrate into a clean dry test tube. Add 4 ml water and than successively 5 ml boric acid, 2 ml ascorbic acid and 1 ml sulfomolybdic acid solution.
Mix and warm for 10 minutes in a water bath at 85°C. Read the absorbance at 665 nm.
Therefore 1 ml of each standard solution is treated exactly in the same way as the soil extracts.

Note : The working series contains 0-10-20-30-40-(50) microgram P.

Remark : Boric acid is added to eliminate the interference of fluoride ions, which have a slight depressive effect on the molybdenum color development.

Reagents

1. Ammonium fluoride stocksolution (approx 2 n). Dissolve 37 g NH_4F in distilled water and dilute to 500 ml Store in polyethylene bottle.

2. Hydrochloric acid stocksolution (approx 0.5 N). Dilute 20.2 ml of concentrated HCl to 500 ml with distilled water.

3. Extracting solution. Add 200 ml of 0.5 n HCl and 15 ml 2 n NH_4F to a 1000 ml volumetric flask and dilute to the mark with distilled water.

4. Ammonium molybdate-sulphuric acid solution. Bring 25 g $(NH_4)_6MoO_{24}.4H_2O$ in a 250 ml beaker and dissolve in 100ml distilled water. Bring 200 ml concentrated H_2SO_4 in a 1000 ml volumetric flask and dilute carefully with 300 ml distilled water. Cool the mixture. Slowly pour the molybdate solution in the acid mixture. Dilute to 1000ml after the combined solutions have cooled to room temperature. Store the solution in the dark.

5. Boric acid solution (0.8 m H_3BO_3). Dissolve 49.4 g H_3BO_3 in distilled water and dilute to 1000 ml.

6. Ascorbic acid : 1 g in 100 ml (keeps for 8 days in refrigerator)

7. Standard P-solutions. From the 100 ppm P solution under 3.1.1.,prepare a series containing 0-10-20-30-40-50 ppm P.

Apparatus :

- 50 ml conical flasks
- test tubes
- pipettes or dispensors for 1,2,4,5ml.

3.2. Extraction and determination of nutrient cations

Procedure

Introduce 5 g air dry soil in a 250 ml erlenmeyer flask and add 100 ml 1.0 n ammoniumacetate (pH 7). Shake for 30 minutes and filter.
In the filtrate K and if necessary, also Mg, Ca and Na can be determined, K, Na and eventually Ca by flame photometry, Mg and Ca (eventually) by atomic absorption. The standard solutions should be prepared in the same 1.0 n ammonium acetate.

Equipment

- 250 ml erlenmeyer flasks
- shaker
- filter funnel stand
- plastic flasks of 125 ml
- atomic absorption spectrometer
- flame photometer

Reagents

- 1.0 n NH_4Ac : dissolve 77.08 g NH_4Ac in 1 litre distilled water

Determination of K, Ca, Na

1. Combined stocksolution :

   2000 ppm Ca : 4.9945 g $CaCO_3$ per litre
   250 ppm Na   : 0.6355 g NaCl per litre
   2000 ppm K   : 3.8133 g KCl per litre.

   Dissolve 4.9945 g $CaCO_3$ in sufficient HCl, add successively the necessary quantities of NaCl and KCl and dilute to 1 litre with distilled water.

2. Calibration standards

mg per litre			ml stock solution	
Ca	Na	K		
0	0	0	0	complete with the extracting solution to 100 ml in volumetric flask
40	5	40	2	
80	10	80	4	
120	15	120	6	
160	20	160	8	
200	25	200	10	

3. Experimental conditions for flame-photometric determination of Ca, K and Na

Flame :

	wavelength in nm	analytical range: ppm in the solution	analytical range : ppm in the soil
calcium	623	0 - 200	0 - 4000
potassium	768	0 - 25	0 - 500
sodium	589	0 - 200	0 - 4000

Remark : Ca can also be determined with atomic absorption spectro-metry. In this case the wavelength is 422.7 nm.

3.3. Determination of available nitrogen

Procedure

Extraction :
Shake 20 g soil in a 200 ml erlenmeyer with 40 ml 1 n KCl for 1 hour and filter

Distillation method :
a) NH_4-N : 15 ml extract, diluted with a little water + 2 drops of phenolphtalein are treated with MgO and immediately distilled. The distillate is collected in a 100 ml erlenmeyer containing 10 ml 3 % boric acid solution. After collection of the ammonia, the borate solution is titrated with 0.01 n HCl.

b) NO_3-N : add a spoonful Devarda alloy ($\pm$2g) to the residue in the distillation flask. The nitrates are reduced to NH_4-N, distilled and determined by titration with 0.01 n HCl.

c) Results :
- NH_4-N : n ml 0.01 n HCl for the titration of the ammonium borate solution, correspond with a NH_4-N content of

$$\frac{n \times 0.01 \times 14 \times 100}{7.5} \text{ mg N per 100 g soil}$$

  Indeed, 15 ml extract correspond with 7,5 g soil.
- NO_3-N : after conversion to NH_4-N
- The N-content is calculated in the same way.

Equipment

- steam distillation apparatus
- 250 ml erlenmeyer flasks
- burette and pipette

Reagents

- 1 n KCl : 74.5 g per litre
- phenolphtalein indicator : 0.1 % in ethanol (70 %)
- MgO powder
- Devarda alloy : powder G.R., Merck 5341
- Indicator : a mixture of equal volumes of methylred 0.66 °/°° and bromocresolgreen (0.99 °/°°) in ethanol (95 %)
- Boric acid 2 % in distilled water containing 10 ml indicator per litre
- 0.01 n HCl

Potentiometric methods

a) NH_4-N
Soil extract : 20 g of air dry soil are shaken in a 200 ml erlenmeyer flask with 40 ml 1 n KCl for 1 hour and filtered in a 100 ml beaker. Add 1 ml 10 m NaOH (pH has to exceed 11). Immerse immediately the NH_3 electrode in the solution and record the potential reading.

b) NO_3-N
Soil extract : 30 g cf air dry soil are shaken with 60 ml 1 % $KAl(SO_4)_2$ solution for 1 hour and filtered into a 100 ml beaker. Immerse the specific nitrate electrode into the solution and record the potential difference against a $Hg/HgSO_4$ reference electrode.

Calculation : a ppm N in the extract correspond with $\frac{a}{5}$ mg N per 100 g soil.

Equipment

- pH meter digital
- NH_3-specific Ion electrode
- magnetic stirrer
- 100 ml beakers
- semi-logaritmic graph paper

Reagents : NH_4-N

- 1 n KCl : 74.55 g KCl per 1
- 10 m NaOH : 400 g NaOH per 1
- 1000 ppm stock solution : dissolve 3.8178 g NH_4Cl in 1 l 1 n KCl. Prepare a standard series containing 1 - 5 - 10 and 20 ppm N by dilution with 1 n KCl.

Reagents : NO_3-N

- 1 % $KAl(SO_4)_2$: 18.4 g $KAl(SO_4)_2$.12 aq in 1 l nitrate free water
- 1000 ppm N stock solution : 721.80 mg KNO_3 in exactly 100 ml 1 % $KAl(SO_4)_2$ solution. Prepare a standard series containing 10 - 20 - 40 - 80 and 100 ppm N by appropriate dilution with 1 % $KAl(SO_4)$ solution.

3.4. Extraction and determination of sulphates

Extraction

Soluble sulphates may be extracted by shaking 10 g soil for 30 minutes with 30 ml water or determined in the Am-Acetate extracts used for determination of exchangeable cations. To determine adsorbed sulphates 20 g soil are shaken for 30 minutes with 100 ml 0.016 M KH_2PO_4 (500 mg P/litre). The adsorbed amount is found by substracting the water soluble from the KH_2PO_4 extractable sulphate quantity.

Determination

Turbidimetric method

Pipette 10 ml extract in a 100 ml beaker. Add 1 ml hydroxylamine hydrochloride and 2 ml $BaCl_2$ reagent. Stir with a magnetic stirrer, equilibrate for 1 hour, stir once more and measure the extinction at 400 nm.

Equipment

- 100 ml beakers
- magnetic stirrer
- spectrophotometer

Reagents

- 5 % hydroxylamine hydrochloride solution (freshly prepared just before use)
- $BaCl_2$ reagent : mix 20 ml Tween 20 and 100 ml of a 10 % $BaCl_2$ solution ; equilibrate for 24 hours. This solution is diluted 10 times with 10 % $BaCl_2$ solution just before use.
- 1000 ppm SO_4^{--} stock solution : dissolve 1.4791 g Na_2SO_4 in 1 l distilled water. Dilute the stock solution to 100 ppm and prepare a series of 0 - 100 - 200- 300 and 400 µg SO_4^{--} by pipetting into 100 ml beakers respectively 0 - 1 - 2 - 3 and 4 ml of the 100 ppm SO_4^{--} solution and making up to 10 ml with distilled water. For further preparation follow the procedure starting with the addition of 1 ml hydroxylamine hydrochloride.

3.5. Extraction and determination of chlorides

Extraction :

Soluble chlorides are extracted with water in the presence of gelatine in order to prevent adsorption of Ag^+ ions at the AgCl particles. Mix 20 g air-dry soil with 40 ml H_2O and 10 ml gelatine solution.

Determination

First a titration curve with a standard NaCl solution is made. Therefore 4 ml 0.05 n NaCl, 10 ml gelatine solution and approximately 40 ml H_2O are mixed and titrated with the $AgNO_3$ solution. From the titration curve the potential at the equivalence point is determined.

To determine the exact normality of $AgNO_3$, 4 ml 0.05 n NaCl, 10 ml gelatine solution and 40 ml water are mixed. After bringing the electrodes in the solution, the titration is carried out till the same potential value is reached.

For the determination of the chloride content of the sample, mix 20 g air-dry soil with 40 ml H_2O and 10 ml gelatine solution and titrate the suspension as described before (a ml 0.05 n $AgNO_3$).

Equipment

- potentiometric titration apparatus
- Ag electrode
- $Hg/HgSO_4$ reference electrode

Reagents

- NaCl 0.05 n = 2.923 g NaCl/l (dried at 400°C)
- $AgNO_3$ 0.05 n = 8.495 g $AgNO_3$/l
- gelatine solution : dissolve carefully 1 g gelatine powder in 200 ml 0.1 n H_2SO_4 at 90°C. After cooling, transfer the solution to a 1 l volumetric flask and bring to the final volume with 0.1 n H_2SO_4.

3.6. Extraction and determination of trace elements

3.6.1. The different extracting solutions for trace elements mentioned in part
1 (n° 3.6) are prepared as follows :

a- 0.5 n NH_4Ac + 0.02 m EDTA[*] at pH 4.65 : dissolve 38.5 g ammoniumace-
tate in 500 ml H_2O + 25 ml acetic acid, add 5.845 g EDTA and bring
the volume to 1 litre with distilled water. Check pH value of 4.65

b- 0.5 n HNO_3 : dilute concentrated HNO_3 (d = 1.4 or $\pm$ 14 E) about 28
times and standardize against a known base solution.

c- DTPA-extractant (0.005 m DTPA, 0.01 m $CaCl_2$ and 0.1 m TEA adjusted
at pH 7.30)
To prepare 10´l of this solution dissolve 149.2 g of reagent grade
TEA, 19.67 g of DTPA and 14.7 g of $CaCl_2.2H_2O$ in approximately 200
ml of distilled water. Allow sufficient time for the DTPA to dissolve
and dilute to approximately 9 litres. Adjust the pH to 7.30 $\pm$ 0.05
with HCl (1:1) while stirring and dilute to 10 litres. This solution
is stable for several months.

d- 1 n NH_4-Acetate (77 g per litre) for Mo-extraction

3.6.2. Extracting procedures

- Solvents a, b and d :
Place 20 g air-dry soil in a 300 ml flask and add 100 ml extracting so-
lution. After shaking mechanically for 30 minutes, filter the suspension
and collect the filtrate in a polyethylene flask. For organic soils a
soil/solution ratio of 1/20 is used.

- Solvent c :
Lindsay and Norvell (26) use a soil/solution ratio of 1/2 (10 g air-
dried soil with 20 ml DTPA extracting solution) and a shaking time of
2 hours. Then the suspensions are filtered through a Whatman 42 fil-
ter paper.
- Hot water extraction for Boron : boil 20 g air dry soil with 40 ml dis-
tilled water in a conical flask with reflux during 5 min. Filtrate after

3.6.3. Determinations   cooling.

Atomic absorption spectrometry with air-acetylene flame is a satisfying
method for the trace elements under consideration Fe, Mn, Zn,Cu and
possibly Co.

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[*] EDTA : acid form $[C_2H_4N_2(CH_2COOH)_4]$

Table 4 gives selected wavelengths for these elements and indicates useful analytical ranges.

Table 4. Analytical data for trace element determinations by atomic absorption

elements	wavelength in nm	analytical range (ppm in the solution)	sensitivity : ppm of elements for 1 % absorption
Iron	248.3	0 - 10	0.048
Manganese	279.5	0 - 6	0.022
Zinc	213.9	0 - 3	0.008
Copper	324.7	0 - 10	0.030
Cobalt	240.7	0 - 4	0.050

Oxidizing flame for all elements.

-Stock solutions and calibration standards :

Stock solutions containing 1000 ppm of the elements are prepared as follows :

Fe - 0.4979 g $FeSO_4.7H_2O$ per 100 ml 1 n HNO_3
Mn - 0.2876 g $KMnO_4$ per 100 ml 1 n HNO_3
Zn - 0.4399 g $ZnSO_4.7$ aq per 100 ml 1 n HNO_3
Cu - 0.3930 g $CuSO_4.5$ aq per 100 ml 1 n HNO_3

Co - 0.4938 g $Co(NO_3).6$ aq per 100 ml 1 n HNO_3

Combined standard solutions containing the desired number of elements may be prepared from these stock solutions. Thus the following master standard can be made :

final concentration	volume of stock solution	
150 ppm Fe	15 ml	mix together in
100 ppm Mn	10 ml	a 100 ml volumetric
50 ppm Zn	5 ml	flask and dilute
100 ppm Cu	10 ml	to 100 ml with
50 ppm Co	5 ml	1 n HNO_3

- Standard series for calibration are obtained by the following dilutions :

ppm in solution					ml of combined standard solutions	
Fe	Mn	Zn	Cu	Co		
0	0	0	0	0	0	dilute to 100 ml with the extracting solution
0.75	0.5	0.25	0.5	0.25	0.5	
1.5	1	0.5	1	0.5	1	
3	2	1	2	1	2	
6	4	2	4	2	4	

Determination of boron

Procedure :

Introduce 4 ml extract in a test tube, add 1 ml buffer solution and 1 ml azomethine H.
Shake and read extinction after 1 hour at 410 nm.

Reagents

-Azomethine H, to be prepared as follows : dissolve 10 g monosodium 4-amino-5 hydroxy napthalein disulfonic acid (Merck Suchardt n° 820078 AM 102) in 500 ml distilled water.
Bring to pH 7 with 10 % NaOH ($\pm$ 10,3 ml) and acidify again with conc. HCl till pH 1.5 ($\pm$ 3,8 ml). Then add 10 ml salicylaldehyde, shake vigorously and warm at 40° to 50°C during 1 hour. After resting overnight filtrate on a porcelain filter G3 and wash the precipitate 5 times with 96 % ethanol, till the filtrate is colourless. Dry the obtained product at 100°C during 3 hours and keep it in a dessicator.
-Azomethine solution : dissolve 0,9 g azomethine H and 2 g ascorbic acid in 100 ml H_2O. This solution must be prepared freshly every day.

-buffer solution : dissolve 250 g NH_4-
Acetate in 500 ml H_2O and add successively
125 ml Acetic acid 99 %
6,7 g Na_2EDTA
6 ml thioglycollic acid 80 %

-1n H_2SO_4

Remark : The B-reagents must be conserved
in polyethylene flasks.

3.7. Simultaneous extraction of major and trace elements (Method of Soltanpour and SCHWAB (36)

Procedure

Ten grams of soil are weighed into
a 250 ml erlenmeyer flask and 20
ml extracting solution added. The
mixture is shaken on an Eberbach
reciprocal shaker for 15 minutes at
180 cycles/minute with flasks kept
open. The extracts are then filtered
through Watman 42 filter paper.

In the extracts the nitrates, phos-
phates, K, Fe, Mn, Zn and Cu are
determined by the normal methods.

Equipment and reagents

A 0.005 M DTPA solution is obtained
by adding 1.97 g DTPA to 800 ml
water. Approximately 2 ml of 1:1
NH_4OH is added to facilitate disso-
lution and to prevent effervescence
when the bicarbonate is added. When
most of the DTPA is dissolved, 79.06
g NH_4HCO_3 (one mole) are added and
stirred gently until dissolved.
The pH is adjusted to 7.6 with ammo-
nium hydroxide. The solution is dilu-
ted to 1.0 litre with water, and is
either used immediately or stored
under mineral oil. The solution is
unstable with regard to pH. However,
if the solution is stored under about
3 cms of mineral oil, the pH remains
fairly stable for two weeks.

B. METHODS OF PLANT ANALYSIS

Plant samples are treated separately in order to prevent any contamination with soil particles. Facilities for drying and grinding are essential : This necessitates ventilation (air-blow), a large drying oven and a separate grinder.

The first step in mineral plant analysis is destruction of organic matter and dissolution of mineral elements. Dry ashing as well as wet destruction methods are given below. If nitrate-N is to be determined, a seperate extraction is necessary, as well as for Sulfur (sulfate) determinations.

1. Ashing for ash determination and dissolution of mineral elements

Weigh 2 g oven dry matter in a porcelain crucible, pre-ash on a heating plate and ash in a furnace at 450°C until the ash turns white.
Quote the ash content as g ash per kg dry matter.
Ashing is the first step of the procedure for determination of K, Ca, Mg, P, Zn and Cu. The further procedure is as follows :
Transfer the ash quantitatively in a 100 ml beaker by means of 20 ml 1 M HCl and digest on a boiling water bath for 30 minutes (watchglass and glass rod). Filter the suspension on an ash free paper filter into a 100 ml volumetric flask. Wash the filter several times with distilled water and make up to the mark. The final concentration is 0.2 m HCl.

Remark : If more trace elements must be determined it is necessary to treat the ash with HF in order to desintegrate unsoluble silicates which may retain important quantities of these elements.
The procedure is as follows : 2g oven-dry plant material are weighed into a platinum crucible, pre-ashed on an electrical plate and ashed in the furnace at 450°C for 2 hours. After cooling, moisten the ash with 3 ml H_2O and add 1 ml concentrated HCl. Heat gently on an electrical plate until appearance of first fumes. Filter on an ash-free filter into a 100 ml volumetric flask, wash 3 to 4 times with warm water.

Transfer filter and residu again into the platinum crucible, put in the furnace and ash at 550°C for half an hour.

Add carefully 5 ml HF to the cooled ash, evaporate without exceeding 250°C, add 1 ml concentrated HCl, filter and wash with warm water. Filtrate and washwater are collected in the 100 ml volumetric flask, containing the first filtrate. Allow the solution to cool and bring to 100 ml with distilled water.

The final concentration of HCl in the solution is $\pm$ 1 %.

2. Wet destruction (18)

Introduce 0.500 g oven-dry plant material in a 50 ml volumetric flask and digest with 2.5 ml conc. H_2SO_4 on a hotplate at approximately 270°C.
Add repeatedly small quantities of H_2O_2 until the digest remains clear. Cool and dilute to 50 ml with pure water.

During digestion some 0.5 to 1 ml of conc. H_2SO_4 is consumed. Therefore standards are prepared in aqueous solutions containing 3.5 ml conc. H_2SO_4 per 100 ml.

3. Analysis of the plant ash solutions and digests

The analytical methods are fundamentally the same as those applied for analysis of soil extracts. The standard solutions for calibration must be prepared with the same basic composition as the analytical solutions : ash solutions contain 20 ml 1 m HCl per 100 ml and digests 3.5 ml conc. H_2SO_4 per 100 ml.

The following methods are used :
- flame photometry : K, Na (air-propane flame)
                     Ca   (air-acetylene flame)
- atomic absorption spectrometry : Zn
- atomic absorption or colorimetry : Mg, Fe, Mn, Cu
- colorimetry : P and eventually Mg, Fe, Mn, Cu
               N (in H_2SO_4-H_2O_2 digests)

3.1. Flame photometric determination of potassium

After dilution of the original ash solution to 1/5, the K-emission is measured in an air-propane flame at wavelength 768 nm. A calibration curve is made with a standard series of 0-200 ppm K.
Quote g K per kg dry matter.

3.2. Determination of calcium

Calcium is determined by atomic absorption in an air-acetylene flame after addition of Strontium to produce a concentration of 1000 ppm Sr in the analyte solution.

Quote g Ca per kg dry matter.

3.3. Determination of magnesium

Identical as for Calcium. Standard series between 0-1 ppm Mg.

Quote g Mg per kg dry matter.

3.4. Determination of phosphorus

Principle

In presence of V^{5+} and Mo^{6+}, orthophosphates form a yellow coloured phospho-vanado-molybdate complex which shows an optimal absorption at wavelength 430 nm.

Reagents

- Nitrovanadomolybdate reagent is prepared by mixing the following solutions :
    100 ml of a 5 % ammonium molybdate solution
    100 ml of a 0.25 % ammoniumvanadate solution
    100 ml diluted HNO_3 (1/3)
- 5 % ammoniummolybdate solution
  Dissolve 50 g $(NH_4)_6Mo_7O_{24}.4H_2O$ in 500 ml warm distilled water (50°C), transfer quantitatively into a 1 l volumetric flask and make up to the mark, after cooling.
- 0.25 % ammoniumvanadate solution
  Dissolve 2.5 g NH_4VO_3 in 500 ml boiling distilled water, cool and add 20 ml HNO_3 (d = 1.4). Make up to the mark with distilled water.
- Prepare standard series between 0 and 25 ppm P.

Procedure

Pipette 5 ml of the original ash solution into a 50 ml volumetric flask. Add 10 ml nitrovanadomolybdate reagent and make up to the mark with distilled water.

After one hour the absorption is measured at wavelength 430 nm with a spectrophotometer.

Quote g P per kg dry matter.

Remark : In order to simplify the manipulation it is also possible to add successively in a test tube : 1 ml sample solution, 4 ml water and 1 ml vanadate reagent.

3.5. Determination of trace elements

Trace elements are determined by atomic absorption in an air-acetylene flame directly on the ash solutions or digests in an analogous way as in soil extracts.

Quote mg per kg dry matter.

4. Chlorides

Potentiometric determination using a Ag electrode and a $Hg/HgSO_4$ reference electrode.

Reagents

- 0.05 n NaCl : 2.923 g NaCl previously dried at 400°C per litre H_2O
- 0.05 n $AgNO_3$: 8.495 g $AgNO_3$ per 1
- 0.2 n HNO_3 : dilute 15 ml HNO_3 (d = 1.4) in 1 1 H_2O.

Procedure

To calibrate the titrator, record a titration curve by titrating a mixture of 4 ml 0.05 n NaCl and 40 ml 0.2 n HNO_3 with 0.05 n $AgNO_3$ solution. From the titration curve the titration end-point (mV) and the exact normality of the $AgNO_3$ solution are determined. After calibration of the instrument, titrations run automatically.

A suspension of 1 g plant material in 40 ml 0.2 m HNO_3 is titrated and the volume of $AgNO_3$ recorded.

Quote g Cl⁻ per kg dry matter.

Remark

Although the procedure described here suggests the use of an automatic titrator, a normal titration using a potentiometer may be carried out in an analogous way.

5. Total nitrogen

Principle

The N in the sample is converted to ammonium (NH_4^+) by digestion with con-
centrated H_2SO_4 in the presence of salicylic acid and a catalyst mixture.
NH_3 is determined after steamdistillation and capture in an excess boric
acid.

$H_3BO_3 + NH_3 \longrightarrow NH_4H_2BO_3$

Titration with HCl $\quad NH_4H_2BO_3 + H^+ + Cl^- \longrightarrow H_3BO_3 + NH_4^+ + Cl^-$
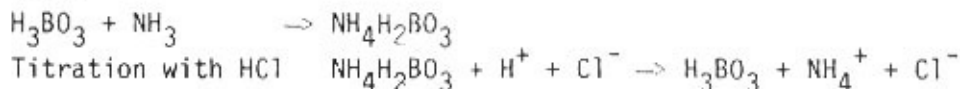
Procedure

Exactly 0.1 g oven-dry plant mate-
rial are treated in a 200 ml Kjeldahl
digestion flask with 10 ml sulfuric
acid - salicylic acid mixture. After
30 minutes, add 5 g $Na_2S_2O_3$ and
shake. After another 15 minutes, add
1.0 g K_2SO_4 and 0.1 g Se as catalysts.
Heat the flask on the digestion rack
for about 3 hours. After cooling, add
30 ml of a 30 % NaOH solution and
start the steamdestillation immedia-
tely, taking care that the glass re-
ceiver tube is immersed into the col-
lecting solution. Collect the distilla-
te into a 250 ml erlenmeyer flask con-
taining 10 ml boric acid-indicator mix-
ture. After distillation of all NH_3,
titrate the boric acid solution with
0.01 n HCl. At the endpoint the indi-
cator turns from green to red.
Quote as g N per kg dry matter.

Reagents

- sulphuric acid - salicylic acid
  mixture : 50g salic.acid in 1 l
  H_2SO_4 (d = 1.84)
- sodium thiosulphate
- potassium sulphate
- selenium powder
- 0.01 n HCl
- 30 % NaOH solution
- indicator : prepare a mixture
  of equal volumes of methylred
  (0.66 °/°°) and bromocresol-
  green (0.99 °/°°) in 95 %
  ethylalcohol
- boric acid-indicator mixture :
  weigh 20 g of H_3BO_3 into a 1 l
  volumetric flask, dissolve in
  600 ml distilled water, add 10
  ml indicator and make up to
  the mark with distilled water.

6. NO_3^--N

Nitrates may be determined after appropriate extraction either by a distillation method or with the specific NO_3^--electrode.

6.1. Destillation method

NO_3^- is reduced to NH_3 by Devarda's alloy and NH_3 is titrated after steam-destillation. Any NH_3 originally present in the sample is previously removed by destillation.

Ammonium and nitrates are extracted from the plant material with a $CaCl_2$ solution, trichloroacetic acid being added to coagulate proteïns.

Reagents

- 2 n $CaCl_2$: 147.03 g $CaCl_2$ in 1 l distilled water
- 10 % and 1 % trichloroacetic acid
- powdered MgO
- Devarda alloy (5 parts Zn, 50 parts Cu, 45 parts Al or Merck n° 5341)
  This alloy is very friable and can be powdered very easily.
- indicator : mix equal volumes of methylred (0.66 °/°°) and bromocresol-green (0.99 °/°°) in 95 % ethylalcohol
- Boric acid - indicator mixture
  20 g H_3BO_3, dissolved in 600 ml distilled water, are mixed with 10 ml indicator and diluted to 1 l with distilled water.

Procedure

a) extraction

Weigh 1 g oven-dry plant material into a 100 ml beaker, add 20 ml 2 n $CaCl_2$ solution and digest on a steam bath for 30 minutes. After cooling add 5 ml 10 % trichloroacetic acid, cover with a watch glass and keep overnight. Transfer quantitatively into a large centrifuge tube, rinsing the beaker with 100 ml 1 % CCl_3COOH.
Centrifuge for 10 minutes (5000 rpm) and filter the supernatant solution into a 200 ml volumetric flask.
Wash the residu in the centrifuge tube twice with 1 % trichloroacetic acid and centrifuge, combine the supernatant solutions and make up to 200 ml.

b) determination

- firstly NH_4^+-N is removed by steam destillation.
Pipette 50 ml of the extract into a 250 ml distillation flask and add 2 drops phenolphtalein indicator. Add MgO powder, just before starting the steamdistillation, until the indicator turns to red.
- after distillation of NH_3 add a spoonfull Devarda alloy to the residu in the distillation flask and distill once more as described before.
Collect the distillate in a 250 ml erlenmeyer flask containing 10 ml boric acid indicator mixture. After distillation titrate the solution with 0.01 n HCl.

Quote result as g nitrate nitrogen per kg dry matter.

6.2. Potentiometric determination of nitrates

Reagents

- extracting solution : 0.04 n $CuSO_4.5H_2O$.
  Weigh 4.936 g $CuSO_4.5H_2O$ in a 1 l volumetric flask, add 1 ml preserving solution and complete to 1 l.
- Preserving solution. 100 mg phenylmercuric acetate and 20 ml dioxane in 100 ml water.
- Standard solution : prepare a standard series containing 2.5 - 5 - 10 - 20 and 25 ppm N.

Extraction

Weigh 1 g oven-dry matter in a polyethylene flask with stopper. Add 25 ml extracting solution and 25 ml water. Shake for 30 minutes and filter into a dry 100 ml beaker.

Determination

Immerse the specific electrode into the solution and record the potential difference against a $Hg/HgSO_4$ reference electrode.
Draw a calibration curve on semi-logarithmic graphpaper. The concentration of N(nitrate) in the sample is read directly from the curve : y mg N per litre.
If v = volume extracting solution and
   p = weihgt of sample

$$mg\ N(NO_3)/kg\ sample = \frac{y \times v}{p}$$

APPENDIX

FORM-MODELS

Laboratory for Soil and Plant Testing

SAMPLE ENTRANCE FORM

Date of sampling :
Laboratory sample number :

- <u>Sampling agent</u> : . . . .

- <u>Farmer</u>   : Name . . . .
            Mailing address . . . .
            . . . .

- <u>Field</u> :   . . . .   <u>Surface</u> : . . . Ha

Soil texture	Drainage	Slope
Sandy : Loamy : Clayey :	Good : Medium : Poor :	Flat : Sloping : Rolling :

General	Past 3 crops	Proposed crop
Irrigation : Salt encrusta- tions : Soil depth : Sampling depth : Stoniness :	19.. : 19.. : 19.. :	1st priority : 2nd priority :

Laboratory for Soil and Plant Testing

SOIL TEST REPORT

Farmer : Name . . . .   Sample number :

Mailing address . . . .   Date :

. . . .

. . . .

Field : . . . .

	value	Estimation		
		low	normal	high
pH–H$_2$O				
pH–KCl				
Phosphorus				
Potassium				
Magnesium				
Calcium				
% carbon				
Salt (mmhos/cm)				
Total nitrogen				
Other :				
sulfate				
zinc				

Fertilizer recommendation for . . . . . (crop)

lime   : . . . kg CaO per Ha

farmyard manure :

nitrogen   : . . . units per Ha

phosphorus   : . . .   "

potassium   : . . .   "

magnesium   : . . .   "

Complementary remarks :

Laboratory for Soil and Plant Testing

SOIL ANALYSIS

Date of arrival :
Date of analysis:

Sampling agent :
Mailing address :

(Listing)

Extracting solution :
Extracting ratio :

Laboratory sample number	pH-H$_2$O	pH-KCl	g/kg dry soil						
			Ca	Mg	K	Na	P		

Laboratory for Soil and Plant Testing

PLANT ANALYSIS

(Listing)

Sampling agent :   Date of arrival :

Mailing address :   Date of analysis :

Laboratory sample number	g/kg dry matter						
	Ca	Mg	K	Na	P		

pH-DETERMINATION

Name of the analyst :   Date of analysis :

Number	pH-H_2O	pH-KCl	pH-$CaCl_2$

Laboratory for Soil and Plant Testing

DETERMINATION OF ORGANIC MATTER

Name of the analyst :

Method of Walkley & Black

Date of analysis :

ml $FeSO_4$ for blanc :

Number	sample weight	ml $K_2Cr_2O_7$ 1 n	ml $FeSO_4$ 1 n	% C	% org. matter

Laboratory for Soil and Plant Testing

FLAME PHOTOMETRIC DETERMINATION

Internal document

Element :

Name of the analyst :

Date of analysis :

Number	dilution	reading	ppm in the dilution	ppm in the extract	g/kg in soil/plant

standards (ppm)					
reading before					
reading after					
average					

Laboratory for Soil and Plant Testing

ATOMIC ABSORPTION

Element :

Name of the analyst

Date of analysis :

Number	dilution	reading	ppm in dilution	ppm in the extract	ppm in plant/ soil	g/kg plant/ soil

standards (ppm)						
reading before						
reading after						
average						

SOIL CONDUCTIVITY AND SALT CONTENT

Name of the analyst :　　　　　　　　　　　　Date of analysis :
Weight of soil : . . . g
Volume water   : . . . ml

Number	Conductivity mmho/cm	salt content in the solution %	soluble salt content %

Laboratory for Soil and Plant Testing

TOTAL NITROGEN DETERMINATION

Name of the analyst :   Date of analysis :

Number	sample weight	ml HCl 0.01 n	mg N/ sample weight	g N/kg soil/plant

Laboratory for Soil and Plant Testing

POTENTIOMETRIC DETERMINATION OF NITRATES

Name of the analyst :   Date of analysis :

Number	reading (mV)	ppm N in the extract	ppm N in soil/plant
Standard serie (ppm)			
reading before (mV)			
reading after (mV)			
Average (mV)			

Laboratory for Soil and Plant Testing

COLORIMETRIC DETERMINATION OF PHOSPHATES

Internal document

Name of the analyst :   Date of analysis :

Number	dilution	reading	ppm P in the dilution	P in ppm in the extract	g P/kg soil/plant
standards in ppm P					
reading					