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FIELD DOCUMENT 3

Soil Mapping and Advisory Services
Botswana

SOIL TESTING PROCEDURES FOR SOIL SURVEY

Part 2
Laboratory Procedure Manual



FOOD & AGRICULTURE
ORGANIZATION OF THE
UNITED NATIONS



UNITED NATIONS
DEVELOPMENT
PROGRAMME



REPUBLIC OF
BOTSWANA

GABORONE, 1988

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Part 2 : Laboratory Procedures Manual

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Food and Agricultural Organization Of The United Nations
United Nations Development Programme

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SAMPLE RECEPTION, GRINDING AND STORAGE

Sample reception:

Incoming samples must always be accompanied by a list of all sample numbers in the lot. Check the samples against this list immediately after arrival.

Report any irregularities to the soil chemist.

The soil chemist or a senior laboratory technician decide which laboratory numbers are to be assigned to the samples.

Record sample numbers and the respective lab numbers in the sample reception book and enter them into the computer data base.

Sample grinding:

Before grinding most samples first have to be dried.

Spread each sample on a tray, together with the sample bag and sample number card. Place in a wind protected spot until dry. Then fill back into bag.

Write lab numbers on the sample containers with a permanent marker. Arrange sample bags and sample containers in serial order to avoid any possibility of mixing up samples.

Fill an appropriate amount of soil from the sample bag into the 'Dynacrush' sample grinder. After about 1 minute switch off. Fill the fine earth fraction which has passed into the receiver through the 2 mm sieve at the bottom of the grinder into the sample container and discard the remainder.

NOTE: Some samples, especially very clay rich samples, form extremely hard aggregates when dry. These aggregates cannot be grinded successfully with the Dynacrush grinder, they might even destroy it. Crush the aggregates first with a stone or a hammer before filling into the grinder. Be careful not to crush any stones.

If no grinder is available crush the aggregates with a rounded piece of wood on a flat tray and pass the soil through a 2 mm sieve.

Store the samples in the sample store room with incrementing lab numbers.

pH-DETERMINATION

Reagents:

For pH-H₂O: distilled water

For pH-CaCl₂: 0.01m CaCl₂ solution

(0.01m CaCl₂ = 1.11 g CaCl₂ per 1 litre or
27.75 g CaCl₂ per 25 liters.)

Buffer solutions: Use pH 7-, pH 4-, pH 9.2-buffer tablets.
Prepare according to instructions on the packet.

Equipment:

Two 25 litre drums for water and CaCl₂-solution.

One pH-meter with electrode. (Zeiss pH-meter 300 with plastic protected 'Orion' electrode 91-55).

Two "AD 1-3 three volume one aliquot" dispensers.

Procedure:

1. Switch the pH-meter in "stand by" position for warming up.
2. Of each sample weigh two times 20g into 100ml plastic bottles. (One set of bottles is marked 'pH-H₂O', the other 'pH-CaCl₂').
3. To the samples in the bottles marked 'pH-H₂O' add 50 ml H₂O dist. with the dispenser connected to the water drum.

To the samples in the bottles marked 'pH-CaCl₂' add 50 ml CaCl₂-solution with the other dispenser.

4. Close bottles tightly, put on shaker in horizontal position and shake for two hours. After shaking remove samples from shaker and let stand for one hour.
5. Switch the pH-meter into 'measuring' mode.
Place the electrode in the pH 7 buffer solution. Wait for stable reading.

(Less than 30 seconds should be required - if it takes much longer the electrode might require cleaning.)

Set the meter to 7.00 with the calibration control.

6. Rinse the electrode with distilled water. Place it in the pH 4 buffer solution (for acid soils), respectively in the pH 9.2 buffer solution (for alkaline soils). Set the meter reading with the slope control.

(The slope should be greater than 92% - if not the electrode might require cleaning.)

7. After calibration immerse electrode into upper part of sample suspension.

Do not stir the suspension.

Wait for a stable reading.

A change of 0.01 units in 10 seconds is considered a stable reading. Note the value, rinse the electrode with distilled water from a wash bottle and continue with the next sample.

8. After pH-H₂O measurement also measure electrical conductivity of this extract.

(To decide whether or not preparation of saturated paste extracts for electric conductivity and/or soluble salts determination is required.)

SATURATED PASTE EXTRACT

Reagent:

distilled water

Equipment:

24-place mechanical vacuum extractor cups for extractor.

large beakers (800-1000ml)

burette (100 ml)

spatula

Preparation of saturated soil paste:

1. Weigh 200 g of soil into beakers
2. With the burette add few ml of water and mix with spatula.
3. Slowly add more water and mix until soil paste glistens as it reflects light, flows slightly when the container is tipped and the paste slides freely and clean off the spatula (for all soils but the soils with high clay content).
4. After mixing allow the soil to stand for one hour or more. Then check saturation criteria again:

If the paste stiffens or loses its glistening appearance remix with water.

Free water should not collect on the surface. If the paste is too wet, add additional dry soil.
5. If soluble salts are to be determined record total amount of water and soil.
6. Cover beakers with watch glasses and let stand for an appropriate time.

Saturated paste extract:

For conductivity determination the extraction can be made a few minutes after preparing the saturated paste.

If the soil contains gypsum the conductivity can increase by 1 or 2 mS/cm upon standing.

Therefore if gypsum is present allow the saturated paste to stand several hours (4-6 hours) before extracting the solution.

If the solution is to be analyzed for its chemical constituents, the saturated paste should stand for 12 hours or more (over night).

Place 9cm fine porous filter paper (i.e Whatman no 42,44 or 50) in extraction cups. Moisten with few drops of water and press it on the bottom of cups.

Hang cups into extractor and connect syringes. Transfer samples into cups. Set extractor to about 1 hour extraction time and start extraction.

ELECTRICAL CONDUCTIVITY

Procedure:

1. Transfer saturated paste extract into small narrow vials. The cell constant of the conductivity electrode, presently in use is 1.00. If other electrode is used check cell constant usually either engraved on electrode or on the connecting cable.
2. Switch the range of the Zeiss con 602 conductivity meter on 'cal' and set the display to 1000 with the 'cal' knob (calibration of cell constant).
3. Record the temperature of the solutions.
4. Switch 'range' into the lowest of the three mS/m positions (do **not** use the μ S/m range).
5. Insert electrode into extract.

NOTE: The two platinum electrodes inside the measuring chamber must be completely immersed in the solution. If the extract is not enough prepare a new saturated paste extract of this sample and combine the two extracts. (this can happen with very clay rich soils).

6. Record the reading and continue with the next sample. If the conductivity is out of the set range, indicated by display of (1.) switch to a higher range.

There are five range settings :

1. 0-2000 micro S/m
2. 0-20.00 mS/m
3. 0-200.0 mS/m
4. 0-2000 mS/m
5. 0-20.00 S/m

As already mentioned above only the three mS/m ranges (2-4) should be used.

If otherwise, it must be clearly indicated in the measuring protocol.

Calculations and data storage:

Data are reported in mS/cm.

To convert mS/m to mS/cm divide reading by 100.

$$\text{mS/cm} = \text{mS/m}/100.$$

Temperature correction:

Use temperature correction table to find correction factor
Figure 1 page 7 (US. Salinity laboratory staff, 1954).

The correction factor also can be calculated with the formula:

$$\text{corr. factor} = 2.581 + (-0.493) * \text{LOG (TEMP).}$$

(Logarithmic regression curve calculated from temperature correction table.)

Soil survey data are calculated and stored with a Hewlett Packard HP 86B Micro computer.

Figure 1: Temperature correction table (US. Salinity Laboratory Staff, 1954)

SOLUBLE SALTS DETERMINATION

Equipment, reagents and standards:

Use same equipment and same chemicals as for exchangeable cations determination.

Take combined stock solutions:

$$\begin{array}{rcl} 200 \text{ meq/l Ca} & + & 50 \text{ meq/l Mg} \\ 50 \text{ meq/l K} & + & 50 \text{ meq/l Na} \end{array}$$

Prepare diluted stock solutions and standard series in the same way as for exchangeable cations determination, only that

the volumetric flasks are filled up with distilled water and not with ammonium acetate solution.

Procedure:

Pipette 1 ml saturated paste extract into 100 ml volumetric flask. Fill up with distilled water and shake well to mix.

NOTE: If absorbance readings of samples are too low with this dilution other ratios can be chosen.

The '1. dilution' then is calculated:

Size of volumetric flask : ml saturated paste extract

Further procedure is exactly as for exchangeable cations determination.

Calculations:

If linear regression curve is calculated concentration C of soluble cations in measuring solution is:

$$C = a * E + b$$

a, b = regression coefficients

E = Extinction (absorbance reading)

If no linear regression curve is calculated concentration C of soluble cations in measuring solution is:

$$C = E / e$$

E = Extinction (absorbance reading)

e = Extinction coefficient

e is calculated from standard curve:

$$e = (E1/C1 + E2/C2 + E3/C3 + \dots + En/Cn) / n$$

$E1, E2, E3, \dots, En$ = Absorbance readings of standards

$C1, C2, C3, \dots, Cn$ = Concentrations of standards

n = Number of standards

Concentration **K** of soluble salts in soil [**meq/l**] is:

$$K = C * \text{extract} / \text{weight} * 1.\text{dilution} * 2.\text{dilution}$$

Concentration **K** of soluble salts in soil [**meq/100 g soil**] is:

$$K = C * \text{extract} / \text{weight} * 1.\text{dilution} * 2.\text{dilution} / 10$$

Extract = ml water used for saturated paste

Weight = g soil used for saturated paste

1. dilution = 100 (1ml extract in 100 ml volumetric flask)

2. dilution = additional dilution

PHOSPHORUS DETERMINATION
(Bray and Kurtz I&II)

Equipment:

Two 25 litre drums for extracting solution and distilled water.
Multi-funnel rack for 33 funnels of approx. 5 cm diameter.
Filter paper Whatman 44 or 42, 9 cm diameter.
Brinkman colorimeter PC 600 / PC 800 with 'dip in' probe or any other colorimeter, preferably with flow through cell.
One AD 1-3 'three volume one aliquot' dispenser or
One AD 3 'three volume three aliquot' dispenser
One SCD 1 'combination diluter-dispenser'
One multisample stirrer

Reagents:

Ammonium Fluoride	NH_4F (MW = 37.05)
Hydrochloric acid conc.	HCl (36%)
Ammonium-hepta-molybdate	$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$
Antimony-Potassium-tartrate	$\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 0.5\text{H}_2\text{O}$
Sulfuric acid conc.	H_2SO_4 (98%)
Ascorbic acid	
Potassium-dihydrogen phosphate	KH_2PO_4

Extracting solution for Bray and Kurtz I:

0.03 m NH_4F in 0.025 m HCl

Dissolve 27.78 g NH_4F in approximately 500 ml distilled/deionized water. Add 53.6 ml HCl conc. (36%). Dilute to 1 litre in volumetric flask, dilute to 25 liters in drum by adding 24 liters distilled/deionized water with measuring cylinders.
Close the drum and mix thoroughly.

Extracting solution for Bray and Kurtz II:

0.03 m NH_4F in 0.1 m HCl

To the dissolved 27.78 g NH_4F add 214.5 ml HCl conc. (36%).

Proceed as above.

Color reagent A:

1. Dissolve in a beaker 30 g of Ammonium-hepta-molybdate, in about 250 ml H₂O dist.
2. Dissolve in a second beaker 0.727 g Antimony- Potassium- tartrate.
3. Fill about 500ml H₂O dist. into a 2000ml volumetric flask.
4. Add **slowly** 340 ml conc. H₂SO₄.
5. Cool in a waterbath to 20° Celsius.
6. Transfer Molybdate and Tartrate solutions.
7. Fill up to the mark with distilled water and transfer into 5 litre bottle.
8. With a measuring cylinder add 3 liters distilled water. Mix well.
9. Store cool and dark.

Color reagent B:

Dissolve 1 g Ascorbic acid per 100 ml of reagent A.

(Approximate requirement for 33 samples plus 5 standards is 250 ml.)

This solution keeps only for one day and has to be prepared new just before use.

Standard solutions for calibration curve:

1. **Stock solution 1000 ppm P/litre:**

Dry Potassium-dihydrogen-phosphate at 80° Celsius in an oven.

Cool in a desiccator.

Weigh 4.393 g and dissolve in 1000 ml volumetric flask with distilled water.

2. **Diluted stock solutions:**

a. 100 ppm P/l:

Pipette 50 ml of 1000 ppm stock solution into 500 ml volumetric flask. Fill with distilled water until about 1 cm below mark. Temperate to 20° Celsius in waterbath and fill up to mark.

b. 10 ppm P/l :

Pipette 50 ml of 100 ppm solution into 500 ml volumetric flask.

Proceed as above.

3. Standard series:

Pipette

10, 20, 30, 40, 50 ml

of the 10 ppm solution into 500 ml volumetric flask.

Proceed as above.

The resulting standard series has the concentrations:

0.2, 0.4, 0.6, 0.8, 1.0 ppm P/I

Settings:

A soil/extractant ratio of 1:7 is used.

Set the dispenser for the extractant to 35ml. Using a AD 3 'three volume-three aliquot dispenser, it must be made sure that all three syringes dispense precisely the same volume.

Set the SCD 1 'combination diluter-dispenser' to dispense 5ml color reagent, 15ml dist. water and take up 20ml extract.

The resulting dilution of 1:2 for samples and standards is found convenient because the absorbance for the highest standard is around 0.2-0.25.

Procedure:

1. Prepare the multi funnel rack. Fold double layers of filter paper and staple the edges that they hold their form. Put the filters into the funnels and place a tray of 33 bottles under the rack.
2. Prepare the color reagent B. For one batch take 250-300ml of reagent A. Add 2.5-3 g Ascorbic acid and stir with a magnetic stirrer until dissolved.
NOTE: Color reagent B is stable only for a few hours and must always be prepared just before the determination.
3. Weigh 5 g of the samples into the bottles of a tray. The last sample should be a standard sample which is repeated with every batch.
4. Quickly dispense 35 ml extracting solution to each of the samples.
5. Stir for 1 minute with the multisample stirrer. (If the Bray and Kurtz II extractant is used stir for 40 seconds.)
6. **Immediately** after stirring filter by pouring simultaneously the contents of 11 bottles (one bottle rack) onto one row of filters. If the filtrates are not clear, filter again using the same filters.
7. Put the tube connected to the syringe calibrated to 5 ml of the combination diluter-dispenser into the color reagent B solution. (The syringe calibrated to 15 ml is permanently connected with the water drum.) Pump at least 5 or more times and discard solution to make sure that all tubes are filled with fresh solution.

8. Place a third bottle rack tray next to the one with the filtrates. Put the double plastic tips of the diluter-dispenser into extract. By pressing the lever take up 20 ml extract, 5 ml color reagent and 15 ml water. Transfer this into the bottles of the third tray by releasing the lever. Dip the tips into a beaker with distilled water to clean the outside.
9. For the standards proceed in the same way. Fill a little more than 20 ml of the standards and extracting solution (for the blanc) into a row of bottles and transfer with the diluter-dispenser into a second row.
10. After 30 minutes to 1 hour the blue color of the molybdate complex is fully developed. With the spectrophotometer determine the absorption of standards and samples at 660 nm wavelength. Use blanc for zero.

NOTE: This standard curve is adapted to the relatively phosphorus-poor soils most widespread in Botswana. Exceptions are diluted with a SD 1 diluter. If the P-concentration is generally higher the standards and the initial dilution (here 1:2) should be changed.

Calculations:

Calculation, storing and printing of soil survey data is done with the Hewlett Packart HP 86B micro computer.

If this is not used the concentration of phosphorus in soil in ppm P is calculated as follows:

Concentration C of P in solution is:

$$C = E / e$$

E = Extinction (absorption)

e = Extinction coefficient

The Extinction coefficient is calculated from the standard curve.

Concentration C of the standards is known. Extinction E is read from the spectrometer.

$$e = (E1 / C1 + E2 / C2 + E3 / C3 + \dots + En / Cn) / n$$

E1, E2, E3, ..., En = Extinction of standard 1, 2, 3, ..., n

C1, C2, C3, ..., Cn = Concentration of standard 1, 2, 3, ..., n

n = Number of standards

ppm P in soil is calculated:

$$\text{ppm P} = C * \text{dilution} * \text{ml Extract} / \text{Weight}$$

NOTE: With dilution is not the initial dilution with the diluter-dispenser meant because there the standards are diluted too.

ORGANIC CARBON DETERMINATION

Equipment:

Conical flasks 50 ml

Drying oven with temperature regulation

Brinkmann PC 600/800 colorimeter with 1 cm light path dipping probe and 570 nm filter or any other spectrophotometer.

acid proof dispenser 0-25 ml

5 litre plastic containers for digestion solution

25 liters drum for Flocculant solution

AD 1-3 'three volume one aliquot' dispensers

SD 1 diluter

bottle rack trays with bottles

NOTE: For dispensing chromic acid the AD 1-3 dispenser proved to be not suitable because the rubber of the plunger was dissolving after some time which caused wrong results.

Reagents:

Sodium dichromate
conc. Sulfuric Acid
Glucose
Superfloc 127

$\text{Na}_2\text{Cr}_2\text{O}_7$
 H_2SO_4
 $\text{C}_6\text{H}_{12}\text{O}_6$

Digestion solution:

Sodium dichromate in approximately 10 n H_2SO_4

In a 5000 ml beaker weigh 745 g of sodium dichromate. Add about 2500 ml of deionized water.

Very slowly and constantly stirring with a magnetic stirrer add 1360 ml conc. H_2SO_4 .

After cooling to 20° Celsius fill up to the 5000 ml mark with water and stir.

(Prepare 10 liters of digestion solution and store them in two 5 litres plastic bottles.)

Standard solutions:

Dissolve:

1.250 g, 2.500 g, 5.000 g, 10.000 g

glucose in 100 ml volumetric flasks with water.

Superfloc 127 Flocculant solution:

Superfloc 127 Flocculant is a nonionic very high molecular weight polyacrylamid. It is highly effective as flocculant in acid, neutral and alkaline suspensions.

Superfloc has been found very effective at a rate of 1 gram per 10 liters of solution.

For 25 l solution weigh 2.5 g Superfloc.

First prepare a concentrated solution in a 1000 ml beaker. Stir 800 ml of water with a magnetic stirrer so that a positive vortex is formed.

Add the dry powder very slowly to the edge of this vortex. (Care must be taken to prevent formation of clots or lumps which then require a very long time to dissolve.)

Continue stirring for one or two hours until all flocculant is dissolved. Stir at less than 400 rpm to prevent breakage of the long Superfloc molecules which would reduce its flocculation power.

Settings:

Set

- the dispenser for digestion solution to 10 ml
- the AD 1-3 dispenser for flocculant to 25 ml
- the SD 1 diluter to dilution ratio of 1:15.(3 ml uptake and 42 ml dispensing.)

Procedure:

1. From each sample weigh 1 g of soil into 50 ml conical flasks.
2. Pipette 1.0 ml of the glucose standard solutions into 50 ml conical flasks. For the blanc take one empty flask where only digestion solution will be filled.
3. Add 10 ml digestion solution.
4. Put the flasks into the oven preheated to 140° Celsius.
5. After 30 minutes take them out and add 25 ml of the Superfloc 127 flocculant solution with the AD 1-3 dispenser.
6. Shake well and let stand for about one hour to allow the particles to settle.

7. With the SD 1 diluter take an aliquot from the clear supernatant solution and transfer into a bottle of the bottle rack tray. (Carefully not to stir the sediment.) Also transfer standards and blanc.
8. Measure absorbance with a Brinkman colorimeter or any other spectrophotometer (preferably with flow through cell) at 570 nm.

Calculations:

For Soil Survey calculation and filing of data is done with the Hewlett Packart HP 86B micro computer.

When 1 g of soil were weighed the standard curve corresponds with:

0.50, 1.00, 2.00, 4.00 % org. C

Example standard 2:

Glucose contains 40 wt% C \Rightarrow 2.50g glucose/100ml = 1g C / 100 ml

1 ml standard = 0.01g C \Rightarrow 0.01g C * 100 / 1g = 1.00 % org. C

NOTE:

If the org. C contents of a sample exceeds 2 %, it must be repeated with 0.5 g of soil.

The standard curve corresponds then with:

1.0, 2.0, 3.0, 4.0, 6.0 % org. C

For samples with more than 6% org C different methods (for example dry combustion method) must be applied.

CEC-DETERMINATION

Equipment:

Automatic Extractor, 24 place, "Concept Engineering, Inc." with:

Syringes, disposable, polypropylene, 60 ml, 'Monoject' reservoir syringes with rubber stopper attached to fit into 'Sample syringes'

Rubber tubing cut in short lengths for connecting syringe barrels

Analytical filter pulp, i.e. Schleicher and Schuell, No. 289

For heavy soils: 'Celite', analytical filter aid or acid washed quartz sand.

Kjeltec distilling unit with storage tank for conc. NaOH-solution

Automatic titration unit (Schott TR 156)

Digestion tubes, 250 ml

Conical flasks, 250 ml

Volumetric flasks, 100 ml

Reagents:

Ammonium acetate

Acetic acid

Ammonia solution

Boric acid

Sodium hydroxide flakes

Volumetric solution of sulfuric- or hydrochloric acid

Ethanol 95% (methylated spirit)

Ethanol 50% (methylated spirit)

Potassium chloride

Hydrochloric acid 36%

1 normal, neutral ammonium acetate solution:

Dissolve 1926.8 g ammonium acetate in 5 liters distilled water.

Make up to 25 liters.

Adjust pH to 7.0 with either acetic acid (if pH > 7.0) or ammonia solution (if pH < 7.0).

1 normal, acidified potassium chloride solution:

Dissolve 1863.9 g potassium chloride (analytical grade) in about 5 l distilled water.

Add 125 ml 36% Hydrochloric acid.

Make up to 25 l.

4% boric acid solution:

Add 200 g of boric acid powder to about 1.5 liters distilled water.

Heat to near boiling and stir until dissolved.

Transfer hot through a large polyethylene funnel into a 5 litre polyethylene bottle containing about 2 liters of distilled water.

Add a few ml of 'mixed indicator' and make up to 5 liters.

Mix well.

Mixed indicator:

Dissolve 0.5 g bromcresol green
and 0.1 g methyl red

in 100 ml 95% alcohol

50% sodium hydroxide solution:

Weigh 2500 g sodium hydroxide flakes in a 5 litre beaker.

Carefully (!) add deionized water up to a volume of 5000 ml.

Stir until dissolved and let cool.

Fill into storage tank of Kjeltac distilling unit. Repeat. (Tank has 10 liters storage capacity.)

NOTE: Fume cupboard, coat, gloves and goggles are imperative!

Sulfuric or hydrochloric acid 0.01 n:

Dilute a volumetric solution accordingly.

(If volumetric solution is 1 normal pipette 10 ml into 1 l volumetric flask and fill with distilled water to about 2 cm below the mark. Put flask in waterbath temperate at 20°C for 30 minutes. Then fill up to mark.)

Procedure:

1. Prepare sample extraction tubes by placing approximately 1 g of filter pulp formed into a ball in a syringe barrel.
Force it down tightly with plunger.
2. Weigh 2.5 g of soil sample. If sample is rich in clay and/or saline, mix well with one 10 ml scoop sand or celite. Fill mixture into tube.
NOTE: A blank should be prepared with every batch. This is imperative if samples have been mixed with sand or celite because both adsorb a certain amount of ammonium which must be subtracted from the result.
3. Place sample tubes in upper disk of extractor and connect to inverted syringe. The plunger of this syringe is inserted in slot of stationary disk of extractor.
4. Fill sample tube to the 15 ml mark with ammonium acetate. Stir carefully to release all enclosed air bubbles from the sample. Rinse the stirring rod and the sides of the tube with ammonium acetate from a wash bottle not exceeding the 25 ml mark.
5. Set extractor to 2 hours extracting time.
6. Add about 45 ml ammonium acetate solution to reservoir tube, start extractor.
7. At the end of extraction the extractor stops automatically by a micro switch. Switch extractor off and pull plungers down as far as extractor will allow.
8. Disconnect syringes from sample tubes (leaving rubber connector on sample tube) and fill contents into numbered 100 ml volumetric flasks.
9. Fill syringes again to 35-45 ml with ammonium acetate solution from a beaker and fill also into volumetric flask. Fill up to volume with ammonium acetate from a wash bottle. Mix well.
10. Keep for exchangeable cations determination.
11. Return upper 2-disc unit to starting position.

12. Attach syringes to sample tubes, rinse sides of sample tubes with ethanol, fill to 20 ml mark.
13. Place reservoir tubes in sample tubes. Fill to about 45 ml mark with ethanol (The total volume of ethanol should be just a little less than 60 ml).
14. After extractor has stopped turn it off, pull plungers down, remove syringes and discard ethanol wash.
15. Fill syringes with acidified 1 m potassium chloride solution and extract. Collect extract in 100 ml volumetric flasks. Fill up to mark with potassium chloride solution and shake well to mix.
Pipette a 50 ml aliquot KCl extract to digestion tubes.
16. Remove sample tubes and transfer samples to the digestion tubes of the distilling unit.
17. Fill 25 ml of the boric acid solution into 250 ml conical flasks.
18. Switch Kjeltac I distilling unit on.
19. Place an empty digestion tube and a beaker in position.
20. Close valve of steam generator outlet at the back of the unit.
21. Open steam valve. Open tap until water level reaches the electrodes in steam generator. Close tap.
22. When water starts boiling open tap again. Regulate water current that the backflowing water (coming from condenser as cooling water) is just hand warm (4-5 liters per minute).
23. Distill about 150 ml into beaker. Close steam valve.
24. Place tube with first sample and first conical flask with boric acid in position. Add 50% sodium hydroxide solution by pulling the lever of the 'alkali pump'.
25. Open steam valve and distill 4-5 minutes (collect 125-150 ml distillate). Close steam valve. Remove tube and conical flask.
26. Place tube with new sample and new conical flask in position. Add sodium hydroxide, open steam valve,
27. During second distillation titrate first sample with 0.01 n H_2SO_4 or HCl and so on.
28. At the endpoint of the titration the green color disappears completely. The solution looks grey. One drop more gives pink. (If an automatic titrator is used, set the end point to pH 4.4.)

Calculations:

CEC in milliequivalent per 100 g soil is calculated as follows:

$$\text{CEC} = (t-b) \cdot N \cdot \text{extract} / \text{weight} \cdot \text{extract} / \text{aliquot}$$

t = ml acid used for titration
b = ml blanc titration
N = normality of acid (= 0.01)
extract = 100 ml
weight = 2.5 g
aliquot = 50 ml

$$\Rightarrow \text{CEC [meq/100g soil]} = (t-b) \cdot 0.01 \cdot 100 / 2.5 \cdot 100 / 50$$

$$\Rightarrow \text{CEC [me/100g soil]} = (t-b) \cdot 0.8$$

EXCHANGEABLE CATIONS DETERMINATION

Equipment:

100 ml volumetric flasks
SD-1 diluter
50 ml polyethylene containers or similar
Varian Atomic Absorption Spectrometer

Reagents:

Strontiumchloride	$\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$	MW = 266.62	AW(Sr) = 87.62	or
Lanthanumchloride	$\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$	MW = 371.37	AW(La) = 138.91	
Calciumcarbonate	CaCO_3	MW = 100.09		
Magnesiumsulfate	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	MW = 246.48		
Sodiumchloride	NaCl	MW = 58.44		
Potassiumchloride	KCl	MW = 74.56		

Interelement effects during Ca and Mg determination:

Interelement effects due to aluminum and silicon and ionization of calcium in the flame might cause problems in the determination. Ionization of calcium and influence of aluminum in the Mg-determination can be reduced by adding strontium- respectively lanthanumchloride to standard- and measuring solutions.

100 000 ppm Sr or La concentrated stock solution

Dissolve 1521.5 g $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ in 5 liters dist. water. (For better solubility add some conc. HCl).
($\text{SrCl}_2 \cdot 6\text{H}_2\text{O} = 32.9\%$ Sr)

or

dissolve 1338.7 g $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ in 5 liters dist. water. (For better solubility add some conc. HCl).
($\text{LaCl}_3 \cdot 7\text{H}_2\text{O} = 37.4\%$ La)

2000 ppm working solution

Pipette 100 ml of concentrated stock solution into 5 liters polyethylene bottle and dilute to 5 liters with distilled water.

Preparation of Ca/Mg standard solutions (meq/litre):

Combined stock solution 200meq/l Ca + 50meq/l Mg

Dry CaCO₃ and MgSO₄*7H₂O at 100° Celsius in drying oven.

Weigh	10.090 g CaCO ₃
and	6.162 g MgSO ₄ *7H ₂ O

into a 1000 ml volumetric flask.

Fill about half with dist. water.

Add some HCl just enough to dissolve the calcium carbonate.

Then fill up to 2 cm below mark and adjust temperature to 20° C in waterbath.

After 30 minutes fill up to mark and shake well.

Diluted Ca/Mg stock solution (1:5)

Pipette 100 ml stock solution into 500 ml volumetric flask.

Fill to about 2 cm below mark with 1 n ammonium acetate solution (from CEC determination).

Adjust temperature to 20° celsius in waterbath.

After 30 minutes fill up to mark, stopper flask and shake well.

This solution contains:

40 meq Ca/l and 10 meq Mg/l.

Standard series

Pipette:

5, 10, 20, 25, 50 ml

of the diluted stock solution into 100 ml volumetric flasks.

Fill to about 1 cm below the mark with 1 n ammonium acetate solution.

Adjust temperature to 20° C in waterbath.

After 30 minutes fill up to mark, stopper flasks and shake well.

The solutions contain:

1. Standard	2 meq/l Ca / 0.5 meq/l Mg
2. Standard	4 meq/l Ca / 1.0 meq/l Mg
3. Standard	8 meq/l Ca / 2.0 meq/l Mg
4. Standard	10 meq/l Ca / 2.5 meq/l Mg
5. Standard	20 meq/l Ca / 5.0 meq/l Mg

Preparation of K/Na standard solutions (meq/litre):

Combined stock solution 50 meq/l K + 50 meq/l Na

Dry KCl and NaCl at 100° Celsius in drying oven.

Weigh 3.7278 g KCl
and 2.9220 g NaCl

into 1000 ml volumetric flask

Fill up to about 2 cm below mark with dist. water.

Adjust temperature to 20° C in waterbath.

After 30 minutes fill to mark, stopper flask and shake well.

Store in polyethylene bottle.

Diluted stock solution (1:25)

Pipette 20 ml of stock solution into 500 ml volumetric flask.

Fill with 1 n ammonium acetate solution (used for CEC determination) to about 2 cm below mark.

Adjust temperature to 20° C in waterbath.

Fill to mark after 30 minutes, stopper flask and shake well.

The solution contains:

2 meq/l K and 2 meq/l Na

Standard series

Pipette:

5, 10, 20, 25, 50 ml

of diluted stock solution into 100 ml volumetric flasks.

Fill to about 1 cm below mark with 1 n ammonium acetate solution (used for CEC determination)

The solutions contain:

1. Standard	0.1 meq/l K + Na
2. Standard	0.2 meq/l K + Na
3. Standard	0.4 meq/l K + Na
4. Standard	0.5 meq/l K + Na
5. Standard	1.0 meq/l K + Na

Alternative K/Na standard solution in ppm:

Combined stock solution 2000 ppm K / 250 ppm Na

Weigh 3.8133 g KCl
and 0.6355 g NaCl

into 1000 ml volumetric flask.

Proceed as above.

Diluted stock solution (1:10)

Pipette 50 ml of stock solution into 500 ml volumetric flask.

Proceed as above.

The solution contains:

200 ppm K and 25 ppm Na

Standard series

Pipette:

5, 10, 15, 20, 50 ml

of diluted stock solution into 200 ml volumetric flasks.

Proceed as above.

The solutions contain:

1. Standard	5 ppm K / 0.625 ppm Na
2. Standard	10 ppm K / 1.25 ppm Na
3. Standard	15 ppm K / 1.875 ppm Na
4. Standard	20 ppm K / 2.5 ppm Na
5. Standard	50 ppm K / 6.25 ppm Na

Ca/Mg and K/Na with Atomic Absorption Spectrometer:

In order to avoid too high absorbencies dilute samples and standards:

Ca/Mg-determination by factor 50
K /Na-determination by factor 10

Connect the SD-1 diluter set to a dilution ratio of 1:50 to the 2000 ppm lanthanum chloride solution. (1 ml sample uptake + 49 ml dispersion.)

Connect the diluter set to a ratio of 1:10 to a drum with distilled water. (5 ml sample uptake + 45 ml dist. water.)

In the literature a 1000 ppm cesium chloride solution is recommended to suppress partial ionization of K and Na. Tests showed that the effect lies within the error range of the determination. The costs for the very expensive chemically pure cesium chloride can be saved.

Dilute ammonium acetate extracts from the CEC determination and standards into polyethylene containers. For blanc dilute ammonium acetate solution, Swi containers to mix. Then fill solutions into vials fitting the automatic sample changer.

Preparation of the Varian AA1275 Atomic Absorption Spectrometer:

1. Setting of acetylene gas pressure:

- a. Slowly open main valve of gas cylinder.
- b. Check that main pressure is more than 700 kpa. (If pressure is below exchange gas cylinder because acetone may be formed and can come into the system.)
- c. Check that gas pressure into system is 70 kpa. (At lower or higher pressure flame might not get started)

2. Operating modes:

The Varian Atomic Absorption Spectrometer can be operated in several modes.

- a. Fully automatic with automatic sample changer and the Hewlett Packart HP86B micro computer as controller and receiver of the data.
- b. Half automatic without sample changer but controlling and data handling by the computer.
- c. Half automatic with sample changer but without computer.
- d. Manual without sample changer and computer.

a. Full automatic operating mode:

Switch system on in the following sequence:

1. Uninterruptable power supply
2. Spectrometer
3. Compressor and exhaust fan
4. Sample changer
5. Printer (make sure that printer is connected to spectrometer and **not** to computer !)
6. Monitor
7. Disc drive
8. Insert HP86/AA1275 - system disc into drive 0 and data disk into drive 1
9. computer

The 'Autostart' program will start up computer. For the further procedure follow instructions displayed on monitor.

In the 'Master Index' section press '4' to enter sample labels. Then press '2' to operate system.

In the 'Optimize Instrument' section select lamp and set wavelength of element to measure.

Set:	Ca-wavelength	(422.6 nm)
	Mg-wavelength	(285.2 nm)
	K -wavelength	(766.5 nm)
	Na-wavelength	(589.0 nm)

NOTE: Lamp alignment must be checked regularly by senior technician.

Enter 'Y' at the question 'Do you want auto set up?'

Enter '1' to have recommended standard settings for spectrometer and sample changer programmed. (In this programme section the keyboards of spectrometer and sample changer are unlocked so that both could also be programmed manually.)

Recommended standard settings: -

Spectrometer:

Lamp 1 (Ca/Mg) : 5 mA
Lamp 2 (K /Na) : 10 mA
Integration time: 1 sec

Absorbance mode (ABS)
Integration hold

Sample changer:

No. standards: 5
Rinse rate : 0
Rinse time : 5
Delay time : 5
Multiples : 4
Reslope rate : 6

Continue following instructions displayed on monitor.

Press 'FLAME ON'. Flame must be light blue to colorless with a sharp light blue rim at the burner. If there are edges in the flame switch off and clean burner slit with a brass blade.

Check whether highest standard is within measuring range. If not, tilt burner until needle moves into leftmost field of peakmeter. (This is necessary for Magnesium using the combined Ca/Mg-standards).

Start automatic run. The end of a run is signalled by a continuous beep from the spectrometer. The system automatically resets. Prepare determination of next element.

NOTE: The setting of the system parameters does not have to be repeated. Press 'N' or 'Spacebar' at the question in the 'Optimize Instrument' section.

After determination of all elements go to 'Master Index' section. Press '6' (transfer data program) to transfer the data into the soil survey data base.

b. Half automatic operating mode without sample changer but with computer as controller:

The same software which controls the full automatic operating mode also handles the half automatic mode without sample changer. The setting of the spectrometer is done either manually or by software using the same recommended parameters as above.

Change samples by hand. To take readings press 'Spacebar', to proceed to next sample press 'CONT'.

Transfer data into soil survey data base the same way as above.

c. Half automatic operating mode without computer but with sample changer:

Make sure that sample changer is in 'Auto sample' mode and not in 'Single sample' mode.

Loop through parameters listing pressing repeatedly 'Programm set up', change parameters using numeric keyboard.

Set spectrometer to recommended parameters. (Spectrometer must be in 'ABS'-mode.)

Load sample changer with samples.

Press 'Start' to start run.

d. Manual operating mode without computer and sample changer:

Operating the spectrometer manually offers a variety of measuring modes:

Readings are displayed and printed either in absorbance or in concentration mode. In concentration mode three standards are allowed from which the concentration of the samples is calculated.

The samples are measured either in 'Integration hold' or in 'Run mean' mode. In 'integration hold' mode impulses are integrated over a certain set time. In 'run mean' mode an accumulative mean is calculated of readings taken between start reading and pressing the 'read' key a second time to end.

For further information refer to the operating handbook for the spectrometer.

Calculations:

In case only absorbencies have been measured and no linear regression curve of the standards can be calculated concentrations in meq/100 g soil are calculated as follows:

Dimension of standards is : **meq / liter**

The concentration C in meq/l of an element in a sample is:

$$C = E / e$$

E = Extinction (absorption)

e = Extinction coefficient

The extinction coefficient is calculated from the standard curve:

$$e = (E1/C1 + E2/C2 + E3/C3 + \dots + En/Cn) / n$$

E1, E2, E3, ... , En = Absorbance readings of standards

C1, C2, C3, ... , Cn = Concentrations of standards

n = Number of standards

The concentration K of a sample in meq / 100 g soil is:

$$K = C * \text{dilution} * \text{extract} / \text{weight} / 10$$

Extract = 100 ml

Weight = 2.5 g

$$K = C * \text{dilution} * 4$$

K/Na determination with 'CORNING 400' flamephotometer:

The emission/concentration relation is linear only up to a certain concentration. Therefore it is advisable to dilute samples and standards for the potassium determination by factor 5.

The concentration of the Na-standards is low enough to stay in the linear range of the emission/concentration function.

Set the SD-1 diluter to a dilution ratio of 1:5 (8 ml solution + 32 ml dist. water).

1. For K-determination dilute ammonium acetate extracts from CEC determinations and standards into plastic containers. For blanc dilute ammonium acetate solution.
2. For Na-determination transfer some of the extracts and standards without dilution into the containers. For blanc use undiluted ammonium acetate solution.
3. Switch power on of the Corning 400 flamephotometer.
4. Open the gas tap. Switch 'Fuel - on'. Open window to the burner chamber and press 'ignition' until flame is burning.
5. Set the filter to 'K' (for potassium).
6. Insert tube into blanc and set to 0 with 'blanc' regulator. Then insert tube into highest standard and set to 100 with 'sensitivity - coarse - fine'. Repeat blanc setting, repeat sensitivity setting until readings are constant.
7. Measure standard curve, then measure samples. Record the values on the appropriate form.
8. When all samples are determined set filter to 'Na' (for sodium) and calibrate the instrument as above (point 6). Measure standard curve and samples and record the values.
9. For closing down first close gas tap, then switch 'fuel - off', then 'power-off'.

PARTICLE SIZE DETERMINATION

Equipment:

1000 ml beakers
watch glasses fitting beakers
rubber policemen for cleaning beakers
1000 ml plastic bottles with tight closing lid
Dispenser for hydrogenperoxide
Dispenser for Na-hexa-meta-phosphate solution
1000 ml measuring cylinders without spout
Stopper to close cylinders
Soil hydrometer(s)

Waterbath with thermostat controlled cooler/heater

Centrifuge equipped to handle samples up to 1000 ml

Sample shaker

Stopwatch

Sieves 53, 106, 250, 500, 1000 μm
Sieve 53 μm for wet sieving
Sieve shaker
Soft brushes for cleaning sieves
A2 paper with smooth surface

2-decimal place balance

Reagents:

Sodium acetate	$\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$
Acetic acid glacial	$\text{CH}_3\text{-COOH}$
Hydrogenperoxide 30%	H_2O_2
Sodium citrate	$\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$
Sodium-bi-carbonate	NaHCO_3
Sodium-di-thionite	$\text{Na}_2\text{S}_2\text{O}_4$
Sodium chloride	NaCl
Acetone	
Sodium-hexa-metaphosphate	$(\text{NaPO}_3)_6$
Sodium carbonate	Na_2CO_3

Preparation of Solutions:

Solutions for pretreatment of samples:

1. **1 normal sodium acetate solution pH 5.0:**

Dissolve 3400 g sodium acetate and dilute to 25 liters in drum. Adjust to pH 5.0 with acetic acid.

2. **0.3 normal sodium citrate solution:**

Dissolve 1305 g sodium citrate and dilute to 25 litres in drum.

3. **1 normal sodium-bi-carbonate solution:**

Dissolve 2100 g sodium-bi-carbonate and dilute to 25 liters in drum.

Solution for dispersion of soil samples:

Sodium-hexa-metaphosphate solution:

Mix 135 g dry sodium-hexa-metaphosphate with 35 g sodium carbonate powder in 5000 ml beaker. Fill up with water and stir until dissolved. Then transfer into storage bottle.

Pretreatment of Samples:

NOTE: Removal of carbonates and free iron-oxides is optional.

1. **Removal of carbonates (if present):(pH > 6.8)**

Weigh depending on the clay contents 50-100 g soil into 800 ml beaker.

Add 250 -500 ml 1 n Na-acetate solution.

Heat on water bath to boiling. After effervescence has stopped, add increments of about 25 ml until it does not recur anymore.

(In case of very high carbonate contents 5 ml increments of glacial acetic acid can be used instead of buffer. In this case monitor the pH with indicator paper.)

Add 500 ml of water and add a few ml saturated NaCl solution for precipitation.

Let stand over-night and siphon off or centrifuge.

2. Removal of soluble salts:

pH > 8.2 EC > 4 mS/cm (400 mS/m)

If no carbonates are present but the electrical conductivity exceeds 4 mS/cm, respectively 400 mS/m soluble salts should be washed out:

Add approximately 200 ml Water to the samples, let stand for about two hours, stir occasionally.

For precipitation add few ml saturated sodium chloride solution or acetone.

Centrifuge excess water, then continue with normal procedure.

3. Oxidation of organic matter:

Only if the org.C contents is bigger than 0.3 % organic matter must be removed.

Add 15 ml water and 15 ml of H₂O₂ (30%) (In case of buffer pretreatment less or no water need to be added). Let stand for 1 or 2 hours.

Place on hotplate and warm to about 50° Celsius.

Add small increments (5-10 ml) of 30% hydrogen peroxide until decomposition of organic matter is completed.

NOTE: Too strong frothing can be tempered by removing from hotplate (cooling in cold water) or adding few drops of alcohol.

In general supernatant solution looks clear after decomposition of organic matter.

To remove the remaining hydrogen peroxide add about 300 ml of water and boil until the volume is reduced to about 100 ml.

If no iron coatings are to be removed, transfer samples into shaking bottles.

4. Removal of iron oxides:

Add 500 ml 0.3 n Na-citrate and 70 ml 1 n Na-bicarbonate solution.

Warm the suspension to 80° Celsius then add 2-10 ml scoops of solid sodium-di-thionite.

Stir suspension constantly for 1 minute and occasionally for 15 minutes. (Avoid heating above 80° Celsius because iron sulfite (FeS) forms.)

Add 50 ml saturated NaCl solution to flocculate.

(If suspension fails to flocculate, add 50 ml acetone.)

Siphon supernatant solution or centrifuge.

Wash with Na-citrate (add NaCl or acetone if necessary for flocculation.)

Siphon or centrifuge again, then transfer samples into shaking bottles.

Procedure:

1. Dispersion of soil particles:

After transferring samples into 1 l shaking bottles add 50 ml sodium-hexametaphosphate solution. Make up volume to about 400 ml to 500 ml with water. (Shaking bottles must be half full to ensure proper dispersion. Do not over fill.)

Put samples on shaker and shake for about 16 hours (overnight) at a speed of about 125 strokes per minute.

2. Clay and silt determination by hydrometer method:

Transfer samples quantitatively from shaking bottles into 1 l measuring cylinders. Fill up to 1 l with water and place in water bath temperature to 20° Celsius. Let stand for about 30 minutes to 1 hour to equilibrate temperature.

Take hydrometer readings for a particular sample after:

40 seconds, 4 minutes, 4 hours

Close the first cylinder with rubber stopper. Shake vigorously end over end for about 1 minute. Make sure that the sample is completely dispersed.

Put cylinder back into waterbath, start stopwatch insert hydrometer carefully take 1. reading after 40 sec.

Close second cylinder, shake and put back into waterbath after precisely 2 min total time on stopwatch. Insert hydrometer and read after 2 min 40 sec total time.

Wait until 4 min take 2. reading of 1. sample. Wait until 6 min take 2. reading of second sample. During waiting shake third cylinder already. Put cylinder back into the bath after 8 min total time. Take 1. reading after 8 min 40 sec.

Shake fourth cylinder and insert at 10 min, read at 10 min 40 sec, wait until 12 min, take 2. reading of third sample, wait until 14 min, take 2. reading of fourth sample, and so on.

Take the third reading after 4 hours settling time.

Starting / reading times for 24 samples see table 2.3.

Separation of sand fraction from clay and silt:

Place a 53 micron test sieve in a large funnel into a sink equipped with a 'soil trap' to avoid blockage of the drainage pipes.

Transfer sample from measuring cylinder on to sieve and rinse thoroughly with water until all clay and silt fraction is washed out. (The water jet should not be too strong to avoid loss of sand fraction due to splashing).

Wash sand fraction from sieve into beaker.

Place beaker on hot plate or drying oven to evaporate water.

4. Dry sieving of sand fraction:

Stack five test sieves 53, 106, 250, 500, 1000 μm meshsize and a receiver pan.

Transfer sand fraction onto the top sieve (1000 μm) and put onto sieve shaker.

Shake for about 5 min at an amplitude of 2.5-3 (amplitude scale is on sieve head).

Brush the fractions on separate large sheets of paper with smooth surface.

Clean sieves thoroughly.

(Do not use brass or steel brushes for sieves with meshsize smaller than 500 micron).

Tare beaker then add fractions starting with receiver, then 53 μm , 106 μm ,

Record each weight.

Calculations:

Sand Fraction:

weight 53 μ fraction = weight 53 - receiver
weight 106 μ fraction = weight 106 - weight 53
weight 250 μ fraction = weight 250 - weight 106
weight 500 μ fraction = weight 500 - weight 250
weight 1000 μ fraction = weight 1000 - weight 500

weight% of fraction = weight of fraction x 100 /total weight

Total % sand = sum weight% of all sand fractions

(receiver fraction is considered coarse silt !)

Clay and silt fraction:

Corrected reading = reading - blank + (temp-20) * 0.36

(Temperature correction: 0.36 is an empiric factor used in literature. Since temperature of waterbath is kept at 20° Celsius, temperature correction is 0).

(Total clay + silt) % = 1. corr reading * 100 / total weight.

A correction factor can be calculated to make:

% Sand + % clay + % silt = 100 %

Let: SS = % sand from sieving
HY = (clay + silt) % + weight % of receiver fraction

Corr. factor = (100 - SS) / HY

This correction factor is based on the assumption that the determination of the sand fraction by sieving is 'correct', no sand is lost during the procedures. It equalizes minor reading or rounding errors since it is difficult to read a hydrometer more precisely than one unit. If no soil hydrometers, calibrated in g soil / liter are available, only hydrometers calibrated in specific gravity (g / liter) the factor corrects the readings to % soil.

The correction factor using soil hydrometers should be in the range of 0.9 to 1.1,

- with hydrometers calibrated in specific gravity in the range of 1.71 to 1.75.

Values outside this range indicate errors in the determination.

Let:

- cr1 = 1. corrected reading (40 seconds)
- cr2 = 2. corrected reading (4 minutes)
- cr3 = 3. corrected reading (4 hours)
- wt = sample weight
- wtr = weight % receiver fraction

% coarse silt = $(wtr + (cr1 - cr2) * 100 / wt) * \text{corr. factor.}$

% fine silt = $(cr2 - cr3) * 100 / wt * \text{corr.factor}$

% clay = $cr3 * 100 / wt * \text{corr. factor}$

Table 2: Time table for hydrometer readings

Start stopwatch
Time : 0:00'00"

hr:min'sec"

	Sample 1	Sample 2	Sample 3	Sample 4
shake until:	0"	02'00"	08'00"	10'00"
1. reading	40"	02'40"	08'40"	10'40"
2. reading	04'00"	06'00"	12'00"	14'00"
3. reading	4:00'00"	4:02'00"	4:08'00"	4:10'00"

	Sample 5	Sample 6	Sample 7	Sample 8
shake until	16'00"	18'00"	24'00"	26'00"
1. reading	16'40"	18'40"	24'40"	26'40"
2. reading	20'00"	22'00"	28'00"	30'00"
3. reading	4:16'00"	4:18'00"	4:24'00"	4:26'00"

	Sample 9	Sample 10	Sample 11	Sample 12
shake until	32'00"	34'00"	40'00"	42'00"
1. reading	32'40"	34'40"	40'40"	42'40"
2. reading	36'00"	38'00"	44'00"	46'00"
3. reading	4:32'00"	4:34'00"	4:40'00"	4:42'00"

	Sample 13	Sample 14	Sample 15	Sample 16
shake until	48'00"	50'00"	56'00"	58'00"
1. reading	48'40"	50'40"	56'40"	58'40"
2. reading	52'00"	54'00"	1:00'00"	1:02'00"
3. reading	4:48'00"	4:50'00"	4:56'00"	4:58'00"

	Sample 17	Sample 18	Sample 19	Sample 20
shake until	1:04'00"	1:06'00"	1:08'00"	1:10'00"
1. reading	1:04'40"	1:06'40"	1:08'40"	1:10'40"
2. reading	1:08'00"	1:10'00"	1:12'00"	1:14'00"
3. reading	5:04'00"	5:06'00"	5:08'00"	5:10'00"

	Sample 21	Sample 22	Sample 23	Sample 24
shake until	1:16'00"	1:18'00"	1:24'00"	1:26'00"
1. reading	1:16'40"	1:18'40"	1:24'40"	1:26'40"
2. reading	1:20'40"	1:22'00"	1:28'00"	1:30'00"
3. reading	5:16'00"	5:18'00"	5:24'00"	5:26'00"

USEFUL ADDRESSES

Part of the equipment used in the laboratory is purchased from overseas. In case of restocking spare parts, breakdowns or replacement here some addresses:

Dispensers/diluters, multisample stirrer, superfloc, etc.:

Custom Laboratory Equipment Inc.
P.O. Box 757
205 E. Michigan Avenue

Orange City, Florida 32763
USA

Telephone: 904-775-2322
Telex: none

Contact: Dr. Alen B. Hunter

Superfloc:

American Cyanamid Company
P.O. Box 32787

Charlotte, North Carolina 28232
USA

Automatic extractor:

Centurion International Inc.
P.O. Box 82846
4555 North 48th Street

Lincoln, Nebraska 68501
USA

Telephone: 402-467-4491
Telex: 48-4377 CENTURION LCN

Contact: Debra Hilde

Barnstead water purification system, steam washer, ultrasound bath:

Curtin Matheson Scientific International
9999 Stuebner Airline

Houston, Texas 77038
USA

Telephone: 713-820-9898
Telex: 46-20313

Contact: Williams

Kjeltec distiller:

Tecator AB
P.O. Box 70

S-263 01 Hoeganaes
Sweden

Telephone: 042-42330
Telex: 72 695

Automatic titrator, pH-electrodes, conductivity cell:

Schott Geraete GmbH or Klever instruments
P.O. Box 1130 P.O. Box 483
Im Langgewann 5

D-6238 Hofheim a.Ts Jukskei Park 2158
Fed. Rep. Germany South Africa

Telephone: 06192-8081 Telephone: 011-704-3017/8
Telex: 410471 Telex:

Atomic Absorption Spectrometer:

Varian Techtron Pty. Ltd. SMM Instruments
P.O. Box 38622

Springvale or Booyens 2016
Australia South Africa

Telephone: Telephone: 434-1010
Telex: Telex: 4-85881

Balances:

Sartorius GmbH or Zeiss West Germany
P.O. Box 3243 P.O. Box 1561

D-3400 Goettingen 2000 Johannesburg
Fed. Rep. Germany South Africa

Telephone: 0551-308 289 Telephone: 402-2860
Telex: 96 723 Telex: 4-88346

Contact: Frank Bender

Shakers, sieve shaker, test sieves:

Labotec Pty. Ltd.
P.O. Box 43161

Industria 2042
South Africa

Telephone: 673-4140
Telex: 4-51117

Contact: Sharon Brett

General Laboratory ware and reliable help for various problems:

Quadralab
P.O. Box

Bergvlei
South Africa

Telephone:
Telex:

Contact: Patric Horey

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