THE LABEX PROGRAMME 1979-1992

A project co-financed by DGIS

INTERNATIONAL SOIL REFERENCE AND INFORMATION CENTRE

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Introduction

The LABEX Programme was initiated by ISRIC on recommendation of the Second International Soil Classification Workshop held in Malaysia and Thailand in 1978. The main aims were to cross-check, correlate and standardize analytical methods for soil characterization to facilitate and improve international soil classification and correlation studies which were seriously hampered by the large variability of analytical data. During the first experimental phase (pilot phase) from 1980 to 1984, 20 laboratories participated in the programme, 10 in developing and 10 in developed countries (for List of Participants see Appendix 4). With a grant from the Directorate General for International Cooperation (DGIS) of the Dutch government a second phase (operational phase) was executed lasting from 1985 to 1992 and in which the number of participants increased to over 110 (see Appendix 2), about a third of which residing in industrialized countries which was considered indispensable for dependable results of the round robin data checks.

Pilot phase

The brunt in organizing the first exchange round was taken by Dr. L.P. van Reeuwijk in 1980 and twenty highly motivated laboratories (10 from developing and 10 from industrialized countries) were invited to participate. From the received data during the first pilot phase two technical reports were produced (13, 14).

It became evident that the variability of data was in general alarmingly high but varied with soil parameter concerned. It also became clear that significant improvement was feasible but that even after standardization of methods a relatively high minimum level of in-between laboratory variability would be unavoidable. Estimates of this variability for some major parameters used in soil characterization were: Clay content 11%, CEC of the clay 25%, Base Saturation 10% (15).

Operational phase

During the second phase, now financially supported by the Dutch Government, the Labex Programme developed into a Project which became an important quality tool for many Soil Laboratories in the world. On request of many participants, more soil parameters were introduced in order to exchange analytical results.

With the introduction of personal computers it became easier to handle data and participants were asked to analyze the samples according to their own methods as well as according to the so called "LABEX procedures" (see Appendix 6), i.e. a two-legged approach.

In August 1986 a very successful International Workshop on the project was organized which was attended by more than 60 participants (for a participant's report see Appendix 5). Co-sponsors were FAO, ORSTOM and USAID/SMSS. The discussions during the workshop indicated clearly that the participating soil laboratories were in the first place interested in a check on the quality of their own performance with their present procedures rather than in entering into tedious standardization efforts where they had to use procedures with which they were not familiar. That such standardization is indeed not immediately successful is demonstrated by Table 1 which shows that the coefficient of variation is significantly less when laboratories use their own procedures. This is no doubt due to the fact that it takes time to get acquainted with a new procedure. The proceedings of this workshop are reported in ISRIC Technical Paper no. 13.

Table 1. Coefficients of variation (CV) for some soil parameters analyzed with LABEX-prescribed procedures and participant's own procedures.

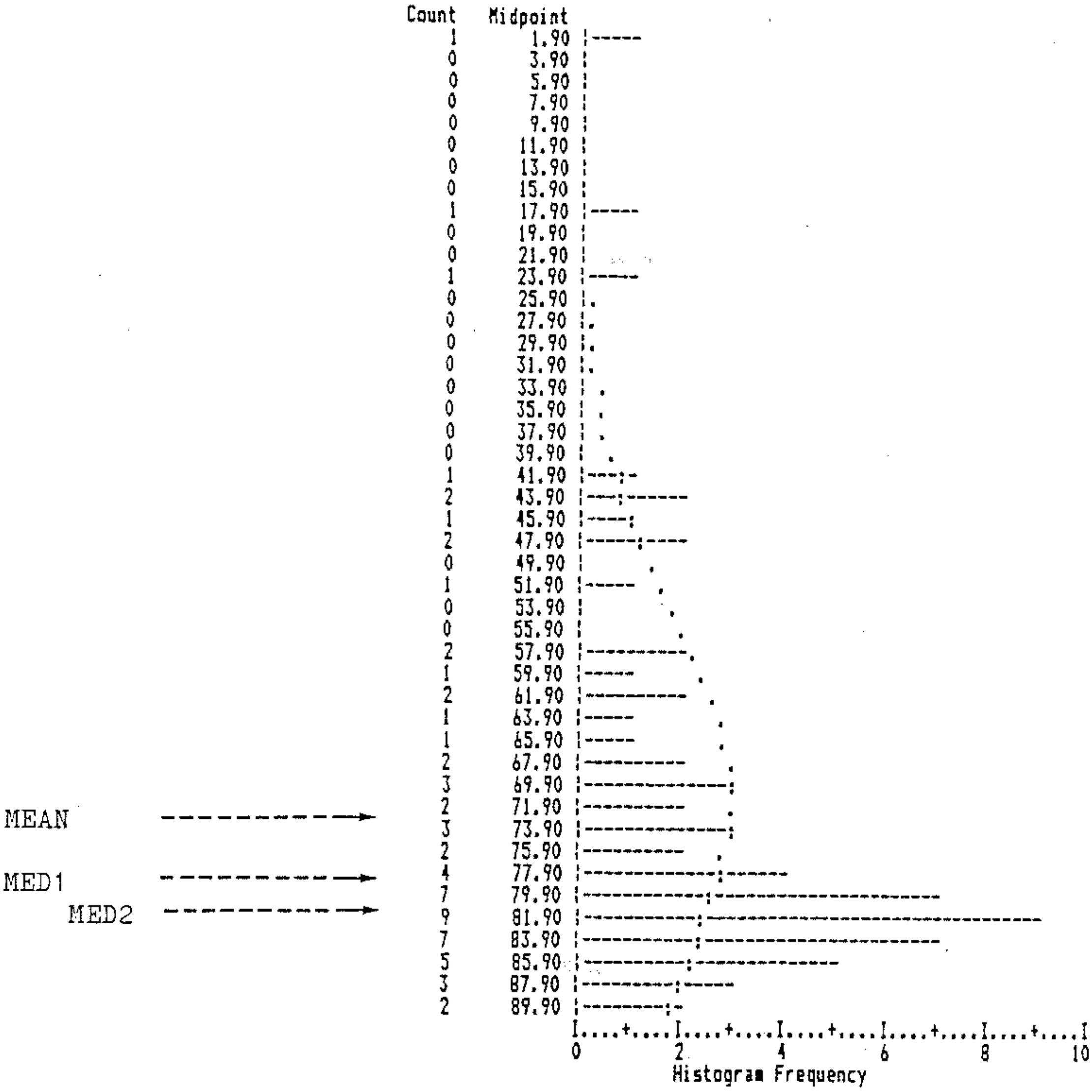
Parameter	%CV LABEX Procedure	%CV own Procedure
pH water	4.25	3.7
pH KCl	2.08	1.0
% Clay	16.62	12.0
% Silt	39.72	12.9
% Sand	61.72	51.9
% Org C	41.01	22.1
CEC	11.98	17.5
Exch. K	8.16	12.7
Exch. Na	20.23	45.3
Exch. Ca	33.10	13.1
Exch. Mg	8.69	17.8
% N	36.70	30.1

In reporting data of such round robin exchange programmes, the identification of outliers, the *median* is preferred to the *average* (Van Montfoort, 1987) so as to reduce the influence of extreme data. After the calculation of the Median (MED1), the Median of the Absolute Differences of the observed values and the median is assessed (MAD1). Values larger than MED1 + $2F \times$ MAD1 or smaller than MED1 - $2F \times$ MAD1 are tagged with two asterisks and are considered as outliers. The same procedure is repeated with the same data excluding those values already tagged with two asterisks. This results in a second median (MED2) and a second Median of the Absolute Differences of the observed values and the second median (MAD2). Values larger then MED2 + $2F \times$ MAD2 or smaller then MED2 - $2F \times$ MAD2 are tagged with one asterisk and are considered as dubious. (The factor F is such that had the data been normally distributed, 5% of the data would have been tagged). The factor F is calculated by:

$$F = (0.7722 + 1.064/n) \times t_{t-1}(0.025)$$

where: n = number of observations
t = the Student T with 0.025% confidence level (n-1 degrees of freedom)

Figure 1 is an illustrative example of a frequency distribution of clay percentages obtained by 66 laboratories for one soil sample (taken from ISRIC Techn. Paper no. 13, p. 11).



Mean = 71.5
MED-1 = 78.5
MAD-1 = 6.5
N = 66 ---▸ F = 1.59

MED-2 = 80.4
MAD-2 = 4.2

Fig.1. Frequency distribution of clay percentages obtained for a round robin sample.

The large variability shown in this diagram is representative for many soil parameters particularly those that involve surface reactions (CEC, extractions) or dispersion (particle-size distribution) and to a much lesser extent for those that involve total contents (carbon, nitrogen). The results of the individual rounds have been laid down in ten technical reports (see Appendix 1) which were distributed among participants.

Special Activities

As a special activity needs to be mentioned the frequent individual attention which was given to those participants that asked for assistance in particular problems. This was done in chiefly two ways: by

correspondence or by personal contact. During the programme period many participants (heads of laboratory or their deputies) visited ISRIC, usually on their way to conferences or training courses. Several did so on repeated occasions.
In addition, ISRIC performed hundreds of analyses (in small batches at a time) on request of participants for checking and reference purposes.
Also, a special round was organized among 8 world-renown soil laboratories to try out four single-step cation-exchange capacity determinations (CEC) as a convenient substitute for the traditional but tedious methods. The silver thiourea (AgTU) procedure appeared to be favoured. This procedure is at present being tested further in several laboratories (also outside the LABEX circle).

LABEX as self-supporting project?

ISRIC was advised by DGIS that financing of LABEX by the latter as of 1991 would be discontinued. In fact, financing of the last three years of the programme would be done on a 100%-75%-50% basis. This prompted ISRIC to investigate the feasibility of self-supporting continued operation of the programme.
For operation as a reference programme only (i.e. without any outreach activity) the following budget was made:

salary professional officer	Hfl. 80.000
sample acquisition and preparation	10.000
postage (samples, reports, correspondence)	15.000
office (housing, computer, stationary)	6.000
printing reports	5.000
	Hfl. 116.000

When 120 laboratories would participate this would imply an annual fee of ca. Hfl. 1000,- per participant. A survey of this "market" with a questionnaire indicated that only about 25 laboratories, predominantly from industrialized countries, would be able to pay this fee[1]. Sponsoring by donor agencies (GTZ, FAO, ORSTOM, SMSS) was explored and resulted in possible commitments of about Hfl. 25.000 per year. Thus, a yearly income of about Hfl. 50.000 would be feasible leaving a deficit of more than Hfl. 65.000 annually. Since this would be a much too heavy financial burden for ISRIC, a non-subsidized continuation of LABEX had to be rejected.

The termination of LABEX financing by the Dutch Government has aroused many protests and expressions of regret by participants for many of whom LABEX was the only outside reference they had. Secondly, the termination of the LABEX programme also implied the discontinuation of a personal approach in case of particular problems arisen in laboratories in developing countries.

Participating laboratories interested in outside references only were advised to participate in the International Soil-Analytical Exchange Programme (ISE) organized by the Wageningen Agricultural University which has many participants (over 200) and a long standing experience particularly with crop analysis. With regard to the discontinuation of "problem solving" assistance, efforts are now made to undertake a programme of improvement of laboratory organization through the introduction of the basic elements of Good Laboratory Practice (GLP) and a Laboratory Information Management System (LIMS). This is discussed next.

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[1] The International Soil-Analytical Exchange Programme of the Wageningen Agricultural University which took over the cross-checking function of LABEX and charging Hfl 500,- per year has great difficulty in finding paying participants in developing countries. This seems not so much due to the underestimation of the importance of participation in round robin cross-checking programmes by heads of laboratories but rather by their financiers.

The Future

An important lesson learned from the Labex project is that the noted variability of analytical data can, amongst other, to a large extent be imputed to essentially two causes:

1. Lack of standardisation of analytical procedures
2. Lack of within-laboratory consistency

Efforts to *standardise procedures* have recently been initiated by working groups of ISO (International Standardisation Organisation) in which ISRIC participates, but the *within-laboratory problems* have thus far been left to its fate.

It is generally accepted that the quality of the output of laboratories strongly depends on the quality of the organization of the work, not only at the level of execution of the analysis but also at management level. To achieve optimal performance, the concept of *"Good Laboratory Practice"* (GLP) was developed and is practised now for quite some time by many categories of laboratories where the quality of the work is of vital importance e.g. in the fields of food, medicine, toxicology, pollution, etc. Implementation of GLP in soil laboratories has not been done on a large scale yet, particularly not in developing countries, but it seems the only way to greatly and structurally improve the laboratory performance. Needless to say that the advantages of this are manifold and invaluable.

GLP prescribes a laboratory to work according to a system of rules which allows better management, improves efficiency (thus reducing costs), minimizes errors, allows quality control (including tracking of errors and their cause) and quality assurance, stimulates and motivates staff, improves safety, improves communication possibilities, both internally and externally. In this way, the performance of a laboratory can be controlled and improved, and the reputation of the laboratory (and that of the whole institute) is improved. An important aspect is also that the pretended quality is documented and can be demonstrated to authorities and clients with the so-called Quality Handbook which contains all the practised measures and rules.

The importance of GLP is illustrated by the observation that these days in many countries governments are introducing the rule to grant orders for analyses only to laboratories that are "certified". For certification the implementation of GLP is an essential requirement. Hence, for our target group we see GLP as a very valuable asset (without aiming at a much more comprehensive certification).

Unfortunately, there is no existing ready-to-use Handbook for GLP in Soil Laboratories. There are, however, guidelines and textbooks which enable laboratories to prepare their own so-called Quality Handbook (which is part of GLP). This documentation (e.g. ISO/IEC Guides no. 25, 38 etc.) is scattered and often difficult to acquire in some countries, not to mention the problems in synthesizing the information into a working manual.

Although all activities for a laboratory can be documented in writing in various note-books (e.g. planning, sample registration etc.) a superior way of doing this is with the help of a (personal) computer. Special programmes for this are commercially available (Laboratory Information Management System: LIMS). However these are generally expensive (>US$ 30.000) and not easy to adapt for soil laboratories. ISRIC has undertaken to develop a LIMS specially for soil laboratories with as major features: user-friendly, easy to adapt to individual laboratory needs, suitable to be used on XT computers, containing several quality control facilities (including some expert system features).

Thus a combination of two virtually tailor-made tools for laboratory organization can become available. The main features can be summed up as follows:

A *Handbook for Good laboratory Practice* (GLP) is an assembly of protocols for a good organization and management of a laboratory.

This includes protocols for the use of

- personnel
- facilities
- equipment
- reagents and materials (including reference materials)
- samples (registration, identification etc.)
- analytical procedures
- calculation procedures and statistical treatment
- quality control
- data entering
- database management
- reporting

A LIMS *(Laboratory information Management System)* is a powerful tool which allows to

- better manage the laboratory
- control GLP in an efficient way (including quality control of data)
- stimulate personnel to apply GLP
- to retrieve and show executed procedures to clients and authorities.

Thus a LIMS handles the *dynamic* part of GLP, the *static* parts being the protocols.

ISRIC intends to prepare this GLP Handbook plus LIMS package, using the relevant guidelines, textbooks and outside consultants as well as our experience in own and developing country laboratories. This package will be made available to good use in upgrading soil laboratories, particularly in developing countries. Since we realize that implementation may in some cases raise practical problems, we have in mind to make available skilled staff that can assist and instruct laboratories for a short period. This would also include the installation and adaptation to local needs of the LIMS. If necessary, this visit can be repeated after a certain period of time. The laboratories themselves should find the finances for this activity.

The remaining stock of bulk samples build up during the LABEX period (see Appendix 3) and representing quite some capital input is considered a very valuable asset and will be used for laboratories in the target group when the GLP/LIMS is implemented.

ISRIC, Wageningen, Summer 1992.

APPENDIX 1. Produced Papers

1. Brunt, J. 1989. A preliminary report of round 1989.

2. Brunt, J. 1990. The laboratory methods and Data Exchange Project. Interim Report on exchange round 1989.

3. Brunt, J. 1990. The laboratory methods and Data Exchange Project. Interim Report on exchange round 1990.

4. Gerits, J. 1989. The Laboratory and Data Exchange Programme. Interim Report on the exchange round 87-88.

5. Pleijsier, L.K., 1985. Interim report on the LABEX exchange round 85-1. ISRIC Working Paper 85/4.

6. Pleijsier, L.K., 1986a. Interim report on the LABEX exchange round 85-2. ISRIC Working Paper 86/1.

7. Pleijsier, L.K., 1986b. Interim report on the LABEX exchange round 86-1. ISRIC Working Paper 86/4.

8. Pleijsier, L.K., 1990. Proceedings of an International Workshop on the Laboratory Methods and Data Exchange Programme.

9. NEWSLETTER 1987-1
10. NEWSLETTER 1987-2
11. NEWSLETTER 1987-3
12. NEWSLETTER 1988

13. Van Reeuwijk, L.P., 1982. Laboratory methods and data exchange program for soil characterization. A report on the pilot round. Part I: CEC and Texture. Techn. Paper 6. ISRIC, Wageningen.

14. Van Reeuwijk, L.P., 1984. Laboratory methods and data exchange program for soil characterization. A report on the pilot round. Part II: Exchangeable Bases, Base Saturation and pH. Techn. Paper 8, ISRIC, Wageningen.

15. Van Reeuwijk, L.P. (1984) On the way to improve international soil classification and correlation: the variability of analytical data. Ann. Rep. 1983, International Soil Museum: 7-14. ISRIC, Wageningen.

APPENDIX 2. Organizations and Institutes participating in the LABEX Programme.

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APPENDIX 3. Types of (bulk) soil samples used in the LABEX project.

1	Kenya	ferric Acrisol A
2	Kenya	ferric Acrisol B
3	Kenya	rhodic Ferralsol A
4	Kenya	rhodic Ferralsol B
5	Kenya	orthic Solonetz A
6	Kenya	humic Nitosol A
7	Kenya	humc Nitosol B
8	Kenya	mollic Andosol A
9	Kenya	mollic Andosol B
10	The Netherlands	calcaric Fluvosol
11	Syria	saline/calc./gypsiferous soil
12	Canada	podzol
13	Malaysia	ferric Acrisol A
14	Malaysia	ferric Acrisol B
15	Hungary	sodic Solonchak A
16	Hungary	sodic Solonchak B
17	Kenya	pellic Vertisol A
18	Kenya	pellic Vertisol B
19	USA	typic Argiustoll A
20	USA	typic Argiustoll B
21	Kenya	humic Andosol A
22	Kenya	humic Andosol B
23	France	mediterranian red soil A
24	France	mediterranian red soil B
25	Brazil	xanthic Ferralsol A
26	Brazil	xanthic Ferralsol B
27	The Netherlands	orthic Luvisol A
28	The Netherlands	orthic Luvisol B
29	Brazil	humic Ferralsol A
30	Brazil	humic Ferralsol B
31	Kenya	orthic Solonetz
32	The Netherlands	Kesteren A
33	The Netherlands	Kesteren B
34	Hungary	Nagykolked A
35	Hungary	Nagykolked B
36	Philippines	pellic Vertisol
37	Mali	ferric Acrisol A
38	Mali	ferric Acrisol B
39	Indonesia	orthic Ferralsol A
40	Indonesia	orthic Ferralsol B

APPENDIX 4

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For our Chief - for your information

APPENDIX 5

TECHNICAL MEMORANDUM 23/1987 CSIRO DIVISION OF SOILS

NOT FOR PUBLICATION

The material contained herein has not been refereed. It may be quoted as a personal communication following written consent of the author.

A REPORT ON THE LABEX WORKSHOP HELD IN WAGENINGEN, THE NETHERLANDS DURING AUGUST 1986

by S. McLeod

INTRODUCTION

For the last 18 months the RAS group in Adelaide have been participating in an interlaboratory programme called LABEX. This programme funded by the Netherlands government through ISRIC in Wageningen was set up with 2 basic objectives (a) to improve soil classification and soil correlation and (b) to provide a reference base for soil laboratories. The prime objective of this programme is directed towards underdeveloped countries by giving them a standard set of soils and data which can be used to calibrate their own methods against international laboratories and to provide a series of optional procedures which are internationally comparable. The programme was to be carried out over a period of 5 years with the majority of participants being from underdeveloped countries. In the first exchange round the laboratories analysed 15 soils for Particle Size (PSA), pH, Exchangeable Cations (EXCATS), Cation Exchange Capacity (CEC) and Organic Carbon (OrgC) using their own methods. In the second exchange round the same soils were analysed for pH, PSA, EXCATS and CEC using the procedures provided by ISRIC. The third exchange round was a repeat of round two using 10 different soils. The results of the first two rounds were circulated to all participants and the third round results were distributed at the workshop. By the end of round three there were 84 laboratories participating in this project throughout the World.

WORKSHOP

The workshop was opened and chaired on the first day by Dr W.G.Sombroek who welcomed the 60 LABEX participants and about 50 observers. The programme secretary Leonard Pleijsier summarised all the LABEX data which showed that as expected the largest variation was obtained with round one when laboratories used their own methods. Rounds two and three also showed a surprisingly high variation considering all laboratories used the recommended procedures. After much discussion it became obvious that a great many laboratories had introduced variations on the "described" procedures through interpretation which it seems was not exclusive to non-English speaking countries. A report was also given on the statistical methods used on the data. In general statistics based on the mean result was thought to be of little value as this only indicated the data's closeness to a mean which may or may not be the correct or a precise result.

The second day was devoted entirely to the determination of CEC and EXCATs. Four papers were given proposing new extractants - Silver Thiourea, Cobalt-Hexamine, Lithium Barium EDTA and Choline Chloride, the latter presented by myself. This paper went over very well and thanks once again to Greg Rinder and the Drawing Office for preparing as always superb slides. These slides were without doubt the best shown at the workshop. Most interest centred on the Silver Thiourea and Choline Chloride procedures and there seemed to be a general desire particularly among the European participants to get away from the USDA Ammonium Acetate procedure. Long discussions followed on the treatment of acid soils (pH 2-4) and calcareous soils (pH8-10) and the merits of prewashing soluble salts or subtracting determined values. This was one of the most interesting days of the workshop.

The third day was split into separate morning and afternoon sessions. During the morning, discussions focused on the results of pH and Org.C determinations. Large variations in pH measurements were again attributed to interpretation of the described procedure as well as types and condition of electrodes, depth of measurement, time after shaking and technique of shaking. The Org.C determinations were then discussed and it appears that most participants used the Walkley and Black or modifications of that procedure. Only a few laboratories had access to a combustion system such as the Leco Analyser and thus it was generally agreed that the better equipped laboratories may be able to help the others by determining a number of their samples by combustion techniques thus enabling them to calibrate their wet digestion method. I then chaired the last session on "Any other Methods" in which I attempted to involve the less vocal "Third World" delegates to talk about their problems in general or specific to the LABEX programme. It would seem that in many cases obtaining a good consistant supply of distilled water of adequate quality was difficult (an additional variation to pH and Cation determinations).

Many had difficulty with soil samples and customs. Quite a number of chemicals of analytical grade were either too expensive or very difficult to obtain and long delivery times were normal. The first afternoon session was an interesting talk from Dr Gene Kamprath of North Carolina on the analytical needs for soil fertility predictions, and an outline of the major soil constituents which LABEX could include. The rest of the afternoon was taken up with laboratory visits.

The fourth day was reserved for Particle Size measurements and general discussions on any of the previous days topics. The large variation of PSA would make any international soil classification system impossible. I feel sure that if the results were reorganised into the various pretreatments a much closer agreement within treatments would result. By a show of hands the great majority of laboratories use a pipette method as their normal routine procedure and as all participants used this method in both rounds 2 and 3, the variations are obviously due not to the method of detection but to the pretreatment leading to the suspension of the soils. Only when precise pretreatment details including any mechanical devices are specified and adhered to by laboratories will there be a significant decrease of this variation. A major break-through in my opinion was the decision to adopt the CSIRO SOILS end-over-end box type shaker as being the cheapest easiest to construct from readily obtained materials, as the standard, for which I have to produce constructional drawings. This means that a completely standard dispersion technique would now be possible. The question then of "what is an adequate dispersion" is an entirely separate problem outside the objectives of the programme which can only be tackled after laboratories prove their ability to reproduce results with an acceptable variability between them.

The last day was probably the most important as this was decision time. The major topics discussed and recommendations made were on the future of the LABEX programme, its format both past and future, financing and liaison between participants. Some of the recommendations were as follows- that the LABEX programme not only continue but be extended to at least 10 years, that the programme should include soil fertility characteristics, that procedures in future will contain precise details and if possible translated into several languages, that some five experienced laboratories be chosen to initially assess new procedures and report on their feasibility before any further inclusion in the programme, that a newsletter be established to further promote mutual exchange between participants, to maintain liaison with the International Standard Organisation (ISO) for which the ISSS have a Standing Committee and that LABEX become a Provisional Working Group under this Committee.

CONCLUSIONS

I thought that this was a most worthwhile workshop and certainly the best I have attended. The informality and venue made one feel at ease and certainly encouraged the exchange of information and ideas at the correct level - the analysts. Many discussions were held spontaneously by smaller groups long after the formal sessions. Thanks must go to Leonard Pleijsier, Peter van Reeuwijk and the Staff of ISRIC for their organisation and management of this workshop. It is their enthusiasm, with the support of the participants, which will go a long way to achieving the goals of this programme. I believe LABEX should be fully supported as the only project today coming to grips with the problems of standardisation of acceptable soil procedures on a truly international basis.

APPENDIX 6

THE LABORATORY METHODS AND DATA EXCHANGE PROGRAMME

LABEX procedures for soil analysis

May 26, 1987

The procedures marked with '*' are attached.

attached	date-code-version			procedure
*	8703	01	1	pH (water, KCl, Ca-chloride)
*	8703	11	1	texture (pipette)
*	8704	12	1	texture (hydrometer)
*	8703	13	1	water dispersable clay
	8704	14	1	aggregate stability
*	8703	19	1	water at 15 bar
	8703	21	1	CEC ammonium acetate
	8703	22	1	CEC choline chloride
	8703	23	1	CEC cobalti hexammine
	8703	24	1	CEC lithium barium edta
	8703	25	1	CEC silver thio urea
	8703	26	1	exchangeable acidity
	8703	27	1	extractable acidity
*	8703	31	1	organic carbon
*	8703	41	1	carbonates
*	8703	42	1	gypsum
	8703	51	1	P-bray I
	8703	52	1	P-olsen
	8703	53	1	P-dabin
	8704	54	1	P-mehlich I
	8703	57	1	P-retention (blakemore)
	8704	58	1	P-retention (north carolina)
	8704	59	1	P-retention (japanese?)
	8703	61	1	dithionite extracted Fe, Al, Mn
	8703	62	1	oxalate extracted Fe, Al, Si
	8703	63	1	pyrophosphate extracted Fe. Al
	8704	64	1	Ca-phosphate extracted S (sulphate)
*	8704	99	1	References

ISRIC P.O. BOX 353-WAGENINGEN-NETHERLANDS

L8703-01-1-1

pH-H_2O, pH-KCl and pH-$CaCl_2$

Principle

The pH of the soil is potentiometrically measured in the supernatant suspension of a 1:2.5 soil:liquid mixture. The liquid is either water, 1 M KCl or 0.01 M $CaCl_2$.

Apparatus

pH meter with appropriate electrode(s)
End-over-end shaking machine
Polythene shaking bottles, 100 ml wide mouth

Reagents

- Potassium chloride solution 1 M.
  Dissolve 74.5 g KCl in water and make to 1 l.
- Calcium chloride solution 0.02 M.
  Dissolve 1.47 g $CaCl_2.2H_2O$ in water and make to 1 l.
- Buffer solutions pH 4.0, 7.0, and 10.0 (or 9.0).

Procedure

Weigh 20 g sample into 100 ml polythene bottle. Include a reference and two blanks.
Add 50 ml liquid with a dispenser or measuring cylinder and cap the bottle.
Shake for 2 hours.
Calibrate pH meter with buffer solutions.
Before opening the bottles for measurement, shake by hand.
Immerse electrode(s) in upper part of suspension.
Read pH when reading has stabilized.
Report also pH values of the blanks.

Note: After pH-H_2O measurement, also measure the electrical-conductivity of this extract to decide whether or not prewashing is required in the exchangeable bases and CEC determinations.

L8703-11-1-1

PARTICLE SIZE ANALYSIS (pipette)

Principle

Two principal steps are involved in particle size distribution analysis: dispersion and fractionation. Dispersion is necessary to separate primary particles from each other and it is performed by the pretreatments. The other step is fractionation. For particles >50 microns sieving is used and for particles <50 micron sedimentation.

Apparatus

Water bath
Hot plate
Reciprocating shaking machine
50 micron sieve
Glass sedimentation cylinders, marked at 1 litre (1 l measuring cylinders can be used)
Drying oven
Tared porcelain dishes
Moisture tins
Pipette 20 ml
Size-scoop 1 g

Reagents

- H_2O_2 30%
- Acetate buffer solution approx. 1 M. Dissolve 680 g Na-Acetate.$3H_2O$ in approx. 4 l water. Adjust to pH 5.0 with approx. 250 ml glacial acetic acid (use pH meter). Make to 5 l.
- Sodium citrate/bicarbonate buffer, 0.3 M citrate and 0.1 M bicarbonate. Dissolve 88 g Na-citrate.$2H_2O$ and 8.4 g $NaHCO_3$ in water and make up to 1 l.
- Sodium dithionite powder
- Dispersion agent: Sodium hexametaphosphate solution 4%, buffered with soda 1%. Dissolve 40.0 g $(NaPO_3)_6$ and 10.0 g Na_2CO_3 in water in a 1 l volumetric flask and make up to volume. Both chemicals should be dried overnight at 105 °C prior to use.
- Sodium chloride, saturated solution
- Sodium chloride 1 M.

Pretreatments

The pretreatments should be applied in the following order:
- oxidation of organic matter
- removal of carbonates
- deferration

The oxidation of organic matter is required for all soils. The carbonate removal is obviously only necessary for calcareous soils. The presence or absence of calcite is assessed as follows:
1. Calcareous soils : pH-H_2O > 6.8
2. Non-calcareous soils : pH-H_2O $<= 6.8$

Deferration is required only if indicated.

Oxidation of organic matter

Weigh out approx. 20 g sample into a 1 l beaker.

Add 15 ml water.

Add 15 ml H_2O_2 30%

Let stand overnight. In case of strong frothing place beaker in basin with cold water. In addition frothing can be tempered by adding a few drops of alcohol.

The next day place beaker on a warm water bath (approx. 80 °C) and regularly add small increments of H_2O_2 30% until decomposition of organic material has been completed (usually supernatant is clear then).

Add water to a volume of approx. 300 ml.

Place on hot plate and boil for 1 hour to remove any remaining H_2O_2.

Remove beaker from hot plate and allow to cool.

Centrifuge and decant or allow material to settle in the beaker and siphon off.

Add approx. 300 ml water and redisperse sediment.

Repeat centrifugation, addition of 300 ml water, and redispersing of sediment until peptization is achieved. Should this take more than 4 washings then add a few ml saturated NaCl solution to promote peptization. With gypsiferous soils many washings are sometimes needed to dissolve all the gypsum.

Removal of carbonate

Add approx. 100 ml acetate buffer solution and heat on water bath (100 °C).

After effervescence has stopped, add increments of approx. 25 ml buffer until effervescence does not recur after addition of new acetate buffer. In case of very high carbonate contents, 5 ml increments of glacial acetic acid can be used instead of buffer. In this case the pH should be monitored with calibrated indicator paper.

Centrifuge and decant or let stand overnight and siphon off the supernatant solution.

Add approx. 250 ml (when centrifuging) or 500 ml (when siphoning) of water and repeat centrifugation or decantation. In case of (partial) peptization, add a few ml saturated NaCl solution.

Deferration

Add approx. 200 ml sodium citrate/bicarbonate buffer solution.

Heat on water bath (approx. 75 °C). Note: Do not exceed 80 °C to prevent precipitation of elemental sulphur.

Add approx. 1 g sodium dithionite powder with a size-scoop and stir constantly for about one minute and then occasionally for 5 minutes with a glass rod.

Repeat twice the addition and stirring of 1 g sodium dithionite powder.

Centrifuge and decant or allow to settle and siphon off.

For samples containing more than 5% extractable Fe_2O_3, repeat the deferration procedure once or twice: a brownish or reddish colour of the sample may indicate still incomplete deferration.

Wash once more with 250 - 500 ml 1 M NaCl.

Dispersion

Transfer suspension quantitatively to a 1 l polythene bottle.

Add 20.00 ml dispersing agent, make the volume to approx. 400 ml water and cap the bottle.

Shake overnight (16 hours) on a end-over-end shaker at approx. 125 strokes/minute.

Separation of fractions

Pass the suspension through a 50 micron sieve which is placed in a funnel positioned above a sedimentation cylinder with a lab stand. Use a small brush or policeman with rubber slap.

Make up to the 1 l mark with water. Note: Include a blank (cylinder with water from same source) for temperature determination.

Sand fraction

Wash the sand fraction remaining on the sieve into a tared porcelain dish, evaporate on waterbath and dry at 105 °C. for about one hour.

Weigh with 0.01 g accuracy (wt. A sand fraction)

Silt and clay fractions

Close the sedimentation cylinder with a rubber stopper and shake well.

Place the cylinder on the table, remove stopper and immediately pipette 20 ml from the centre of the cylinder (see note below).

Transfer the aliquot to a tared moisture tin, dry overnight at 105 °C.

Remove tin from drying oven, close with lid and cool in dessicator. Weigh with 0.001 g accuracy (wt. B for fraction < 50 micron).

Again stopper the cylinder and shake well.

Place the cylinder on a vibration-free table under the pipette-assembly.

After exactly 5 minutes pipette 20 ml at depth indicated in the table.

Transfer aliquot to tared moisture tin, dry overnight at 105 °C.

Remove tin from drying oven, close with lid and cool in dessicator.

Weigh with 0.001 g accuracy (wt. C for fraction < 20 micron).

Again stopper the cylinder and shake well.

Place the cylinder on a vibration-free table under the pipette-assembly.

After 5.5 hours pipette 20 ml at a depth indicated in table 1.

Transfer aliquot to tared moisture tin, evaporate on waterbath and dry overnight at 105 °C.

Weigh with 0.001 g accuracy (wt. D for fraction < 2 microns).

Calculation

The calculation base is the oven dry sample weight after all the treatments. It is obtained by summation of all individual fractions:

clay (<2 micron) = (D * 50) - 1.000 g (wt. K)
silt (2-20 micron) = (C * 50) - 1.000 g - K (wt. L)
silt (20-50 micron) = (B * 50) - 1.000 g - K - L (wt. M)
sand (50-2000 micron) = wt. A

1.000 g = correction for dispersing agent

Sample weight = K + L + M + A
The proportional amounts (in %) of the fractions are now calculated by:

% clay (<2 micron) = (K/sampleweight) * 100
% silt (2-20 micron) = (L/sampleweight) * 100
% silt (20-50 micron) = (M/sampleweight) * 100
% sand (50-2000 micron) = (A/sampleweight) * 100

Note: When a 20 ml pipette is not available, a 25 ml one can be used. In that case the multiplication factor of 50 should be changed into 40, and the correction for dispersing agent becomes 1.250 g.
Before pipetting measure the temperature in the blank cylinder.

Table 1 Depth (in cm) at which fraction <20 micron and <2 micron are pipetted as a function of the temperature and after indicated settling time

Temperature °C	5 mins. <20 micron	5.5 hours <2 micron
15	9.6	6.2
16	9.8	6.4
17	10.1	6.6
18	10.3	6.7
19	10.6	6.9
20	10.9	7.1
21	11.1	7.2
22	11.4	7.4
23	11.7	7.6
24	12.0	7.8
25	12.2	8.0
26	12.4	8.2
27	12.7	8.4
28	13.0	8.6
29	13.3	8.8
30	13.6	9.0
31	13.9	9.1
32	14.2	9.3
33	14.4	9.5
34	14.8	9.7
35	15.1	9.9
36	15.4	10.1

References

P.R. Day in: Black (1965), p. 545
Jackson (1969)
USDA/SCS (1972, 1982)

PARTICLE SIZE ANALYSIS (hydrometer)

Principle

The density of a soil suspension at a given depth becomes less as the particles settle. Its value at different times is related empirically to particle size. The density is measured with a special hydrometer, calibrated directly in percentages.
Dispersion, necessary to separate primary particles from each other, is performed by a treatment with sodium hexametaphosphate. Soils containing much organic or calcareous material should be putted through the other pretreatments as described with the pipette method (8703-11).

Apparatus

Hydrometer set, calibrated in grams per liter
Stirrer (milk-shake type)
Reciprocating shaking machine
Hydrometer jars, marked at 1 litre
Interval timer or stopwatch
Special plunger for mixing, consisting of a circular brass disc, about 55 mm diameter, pierced with 8 to 10 holes of 4 - 5 mm diameter, fastened to a 600 mm length of brass rod.

Reagent

- Dispersion agent: Sodium hexametaphosphate solution 4%, buffered with soda 1%
  Dissolve 40.0 g $(NaPO_3)_6$ and 10.0 g Na_2CO_3 in water in a 1 l volumetric flask and make up to volume. Both chemicals should be dried overnight at 105 °C prior to use.

Dispersion

Transfer 50 g oven dry soil to a 1 l polythene bottle.
Add 100 ml dispersing agent.
Shake overnight (16 hours) on a end-over-end shaker.
Transfer soil and solution to the cup of a mechanical stirrer washing out the bottle and making the volume in the cup to about 500 ml with water.
Stir for 2 - 5 minutes.

Readings

Transfer the dispersed soil suspension to a hydrometer jar, washing out the stirrer cup and adjust the volume in the jar to one liter with water.
Mix and read the temperature.
Mix with the special plunger by moving it up and down in the jar.
Start the timer and after the selected interval, lower the hydrometer carefully in the centre of the suspension and release it gently.
Take the reading on the scale to the nearest 0.5 unit.
Repeat the mixing of suspension and take the next reading after the indicated interval.
Record readings A, B and C after the intervals given in table 1.

Table 1 Settling times

time	Reading
40 sec.	---> reading A
4 min.	---> reading B
2 hrs.	---> reading C

Calculation

The calculation is based on the calibration of the hydrometer in grams soil material per liter in the suspension.
The proportional amounts (in %) of the fractions are calculated by:

% clay(<2 micron) = (C+TC)*mcf/s * 100
% silt(2-20 micron) = (B+TC)*mcf/s * 100 - %clay
% silt(20-50 micron) = (A+TC)*mcf/s * 100 - %clay - %silt(2-20)
% sand(50-2000 micron) = 100 - %clay - %silt(2-20) - %silt(20-50)

in which:

TC = temperature correction (see table 2)
mcf= moisture correction factor ((100+%moisture/100)
s = air dry sample (g)

Note: With differently calibrated hydrometers the calculation should be adapted accordingly.

Table 2 Temperature Correction

Temperature °C	Correction g per litre
15	-2.0
16	-1.5
17	-1.0
18	-1.0
19	-0.5
20	nil
21	+0.5
22	+1.0
23	+1.0
24	+1.5
25	+2.0

References

Gee and Bauder in: Klute (1986), p. 404
Dewis and Freitas (1971)

L8703-13-1-1

WATER DISPERSABLE CLAY

Principle

Water dispersable clay is the clay content found when the sample is not pretreated and when no dispersing agent is added prior to shaking.

Apparatus

Polythene bottles 1 l.
End-over-end shaking machine
Tared moisture tins

Reagents

Procedure

Weigh 10.00 g sample into a 1 l polythene bottle.
Add approx. 400 ml water and shake overnight in shaking machine.
Transfer to a 1 l sedimentation cylinder and make to the mark with water.
After 5 1/2 hours pipette a 20 ml aliquot at a depth indicated in the table below.
Transfer aliquot to tared moisture tin, evaporate on water-bath and dry overnight at 105 °C.
Remove tin from drying oven, close with lid and cool in desiccator.
Weigh with 0.001 g accuracy (net weight = Q g).

Calculation

% clay = Q/s * 50 * mcf * 100%

in which:
s = air dry sample weight in grams
mcf = moisture corection factor ((100+%moisture)/100)

TABLE Depth (in cm) at which fraction < 2 microns is pipetted after 5 1/2 hours settling time, as function of the temperature (°C).

Temp	Depth	Temp	Depth	Temp	Depth	Temp	Depth
19	6.9	24	7.8	29	8.8	33	9.5
20	7.1	25	8.0	30	9.0	34	9.7
21	7.2	26	8.2	31	9.1	35	9.9
22	7.4	27	8.4	32	9.3	36	10.1
23	7.6	28	8.6				

Reference

USDA/SCS (1972, 1982)

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WATER RETENTION AT 15 BAR

Principle

The water content is determined in a soil sample after equilibration with water at 15 bar tension. As the 15 bar water is confined to micro pores a disturbed sample is used.

Apparatus

15 bar pressure plate extractor (Soil Moisture Equipment Corp.) with pressure source and necessary control manifolds
Rubber rings 5 cm diameter, 1 cm high
Tared moisture tins

Procedure

Saturate the porous extractor plates by submerging them in water for 24 hrs.
Place rubber rings on saturated plate.
Fill each ring with approx. 25 g sample.
Add water drop-wise until the sample is just saturated.
With the back of a spoon slightly compress the sample to ensure good contact between soil particles, aggregates and pressure plate. Do not puddle!
Install plates in extractor.
Close extractor and leave for 6 hrs. Then apply pressure according to instruction of manifold. Leave for 1 week. Check and readjust pressure every day.
Note: Inspect outlet of the porous plate. If it continues to bubble after a few hours, the plate is probably defective and should be replaced.
Release pressure and open the extractor.
Transfer sample to a tared moisture tin with a spoon. Weigh immediately with accuracy 0.01 g (net weight = K).
Dry overnight in drying oven at 105 °C.
Remove tin from oven, close with lid and cool in desiccator.
Weigh with 0.01 g accuracy (net weight = L).

Calculation

Moisture content at 15 bar = (K - L)/L * 100% (weight %)

Note: In soil water retention studies the moisture content is usually expressed as volume-percentage. This volume % can be obtained from the weight % by multiplication with the bulk density. As Labex samples are disturbed samples the bulk density is unknown.

References:

Klute in Klute (1986)
USDA,SCS (1972,1982)

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ORGANIC CARBON

Principle

The Walkley Black procedure is followed. This involves a wet combustion of the organic matter with a mixture of potassium dichromate and sulphuric acid. After reaction the excess dichromate is titrated against ferrous sulphate.

Apparatus

Safety pipette 10 ml
Magnetic stirrer (preferably illuminated)

Reagents

- Potassium dichromate standard solution 0.1667 M.
  Dissolve 49.04 $K_2Cr_2O_7$ A.R., dried at 105 °C, in water in a 1 liter volumetric flask and make to volume.
- Concentrated sulphuric acid (96%).
- Concentrated phosphoric acid (85%).
- Barium diphenylamine sulphonate 0.16% (indicator).
  Dissolve 1.6 g barium diphenylamine sulphonate in 1 l water.
- Ferrous sulphate solution 1 M (approx.).
  Dissolve 278 g $FeSO_4.7H_2O$ in approx. 750 ml water and add 15 ml concentrated H_2SO_4. Transfer to a 1 l volumetric flask and make to volume with water.

Procedure

Grind appprox. 5 g sample to pass a 0.5 mm sieve.

Weigh 1.00 g of this material into a 500 ml wide-mouth erlenmeyer flask. Include a reference and two blanks. Note: In case of soils containing more than approx. 2.5% C proportionally less sample should be used.

Add 10.00 ml dichromate solution.

Carefully add 20 ml sulphuric acid with measuring cylinder and swirl the flask and allow to stand on a pad for 30 minutes in a fume cupboard.

Add about 250 ml water and 10 ml phoshporic acid with a measuring cylinder and allow to cool.

Add 1 ml indicator and titrate with ferrous sulphate solution while the mixture is being stirred. Near the end-point the brown colour becomes purple or violet-blue and the titration is slowed down. At the endpoint the colour changes sharply to green. If more than 8 ml ferrous sulphate solution is needed then repeat the determination with less soil.

Note: The end-point is easily overshot; in that case add 0.50 ml of the dichromate solution and titrate again (calculation has to be adapted accordingly).

Calculation

The carbon content of the soil is:

$$\%C = M * 0.39 * mcf * (V1 - V2)/s$$

in which:
M = molarity of ferrous sulphate solution
$V1$ = ml ferrous sulphate solution required for blank
$V2$ = ml ferrous sulphate solution required for sample
s = air dry sample weight in gram
mcf = moisture correction factor ((100+%moisture)/100)
$0.39 = 3 * 0.001 * 100\% * 1.3$ (3 = equivalent weight of C)

Note: The factor 1.3 is a compensation factor for the incomplete combustion of the organic matter. This ineffectiveness varies with the type of organic matter and the factor 1.3 is a compromise.

Reference

D.W. Nelson and L.E. Sommers in: Page (1982) p. 570
USDA. Soil Conservation Service (1982)

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CARBONATE

Principle

The method presented here is the acid neutralization method. The sample is treated with dilute acid and the excess acid, not neutralized by the carbonates, is titrated. The results are referred to as 'calcium carbonate equivalent' since the acid neutralization is not selective for calcite. Other carbonates such as dolomite are also dissolved to some extent.

Apparatus

Burette
Polythene wide mouth shaking bottles
End-over-end shaking machine

Reagents

- Hydrochloric Acid 1 M.
  Add approx. 4 l water to a graduated 5 l beaker or flask, slowly add 425 ml concentrated HCl under constant stirring. Cool and make to 5 l with water.
- Hydrochloric Acid 0.500 M standard solution.
  Dilute standard solution concentrate ampoule according to instruction.
- Sodium hydroxide solution 0.5 M standardized.
  Dissolve 20 g NaOH pellets in 1 l water. Standardize by titration immediately before use against 0.500 M standard HCl using phenolphtalein as indicator.
- Phenolphtalein indicator solution 0.1%.
  Dissolve 100 mg phenolphtalein in 100 ml ethanol 96%.

Procedure

Weigh 5.00 g sample into a shaking bottle. Include two blanks and a reference sample or 500 mg $CaCO_3$ powder.

Note: If expected carbonate content > 50% then use 2.50 g sample.

Add 100 ml 1 M HCl by pipette and swirl.

Loosely screw on lid (do <u>not</u> tighten, so CO_2 can escape!) and swirl occasionally during the next hour. Let stand overnight.

The next day, indent the bottle by hand, tighten the lid and shake for two hours in the shaker.

Let the suspension settle or filter off, pipette 10 ml supernatant solution into a 100 ml erlenmeyer flask and add about 25 ml water.

Add a few drops phenolphtalein indicator and titrate with 0.5 M NaOH.

Calculation

% $CaCO_3$ equivalent = mcf * 50 * M * (a - b)/s

in which:
a = ml NaOH used for blank
b = ml NaOH used for sample
s = air dry sample weight in grams
M = Molarity of NaOH solution
50 = 50 * 0.001 * 10 * 100% (50 = equivalent weight of $CaCO_3$)
mcf = moisture correction factor ((100+%moisture)/100)

Note:
By this method the carbonate content is somewhat overestimated as also non-carbonate contents of the soil may react with the HCl. At very low contents (< 1%) the error could be relatively large. Therefore the analysis is not carried out when pH-H_2O < 6.8 as carbonate is then assumed to be absent.

<u>Reference</u>

L.E. Allison and C.D. Moodie in: Black (1965) part 2, p. 1387
Hesse (1971)

L8703-42-1-1

GYPSUM

Principle

Gypsum is dissolved by shaking the sample with water. It is selctively precipitated from the extract by adding acetone. This precipitate is redissolved in water and the gypsum is determined by measuring the Ca concentration in the solution.

Apparatus

Polythene bottles, wide mouth, 250 ml
End-over-end shaking machine
Centrifuge
Atomic adsorption spectrophotometer

Reagents

- Acetone, analytical grade
- Barium Chloride Solution 1 M.
  Dissolve 60 g $BaCl_2.2H_2O$ in a graduated 250 ml erlenmeyer flask and make to 250 ml 250 ml with water.
- Hydrochloric Acid 1 M.
  Dilute 21 ml concentrated HCl in 200 ml water in a 250 ml graduated erlenmeyer and make to 250 ml with water.
- Nitric Acid 6 M.
  Dilute 375 ml conc. HNO_3 with water to 1 l.
- Lanthanum suppressant stock solution, 1% La
  Dissolve 35.2 g La_2O_3 in 160 ml HNO_3 6 M and dilute with water to 3 l (excess HNO_3 : 0.1 M). (An equivalent solution using $La(NO_3)_3$ and HNO_3 can be made as alternative).
- Standard solution 1000 ppm Ca.
  Dilute a standard analytical concentrate ampoule (1 mg/l) according to instruction.
- Standard series Ca.
  Of the 1000 ppm Ca standard solution pipette 25 ml into a 250 ml volumetric flask. Add 125 ml La suppressant solution and make to volume with water. Of this 100 ppm Ca standard solution pipette 0-5-10-15-20-25 ml into 100 ml volumetric flasks respectively and make to volume with water. The standard series is then 0-5-10-15-20-25 ppm Ca.

Procedure

Weigh 10.0 g sample into a 250 ml polythene bottle. (see Note below). Include a reference sample or 100 mg $CaSO_4.2H_2O$.
Add 100 ml water by pipette.
Cap bottle and shake for 30 min.
Centrifuge the suspension until supernatant is clear.
Test for sulphate:
  Transfer ca. 3 ml extract to a test tube and add approx. 10 drops of 1 M HCl and approx. 2 ml 1 M $BaCl_2$ solution. Only if a turbidity develops gypsum is present and the analysis is continued. Note: For this test the extract of the pH-H_2O determination can be used also.
Pipette 20 ml extract into a 50 ml centrifuge tube. Add 20 ml acetone, mix thoroughly and let stand for 10 mins.
Centrifuge until the supernatant is clear.
Decant the liquid carefully so no precipitate is lost.
Redisperse the precipitate with 10 ml acetone by flowing it from a pipette along the wall of the centrifuge tube.
Centrifuge and decant.

Dry the tube with the precipitate in drying oven at approx. 50 °C leaving the door ajar.
Add 40 ml water by pipette, stopper the tube and shake until the precipitate has dissolved.
Pipette 2 ml of this solution and 2 ml La suppressant solution into a test tube. Homogenize and measure Ca by AAS at a wavelength of 422.7 nm.

Calculation

% gypsum = (reading ppm Ca) * df * mcf * 0.086/s

in which:
df = dilution factor to bring final solution within the measuring range
mcf = moisture correction factor ((100+%moisture)/100)
0.086 = 40/1000 * 0.001 * 100/20 * 172.17/40.08 * 100%
(172.17 = mol wt. gypsum; 40.08 = mol wt. Ca)
s = sample weight in gram

Note:
The solubility of gypsum in water is approx. 0.2 g/100 ml. In the present procedure, this corresponds with 2% gypsum in a sample using a 1:10 soil/water ratio. For practical reasons the maximum content should be set at 1.5%. At higher gypsum contents a proportionally wider soil/water ratio should be used.
e.g. up to 3% gypsum: 5 g sample in 100 ml water (1:20)
up to 4.5% gypsum: 5 g sample in 150 ml water (1:30)
etc.

Reference

Hesse (1971)

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