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NEWSLETTER

NEWSLETTER

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LABEX-01

Labex secretariat:
ISRIC P.O.Box 353
6700 AJ Wageningen
The Netherlands.

This first issue

This is the first issue of the LABEX Newsletter. During the Workshop, last August, many participants were in favour of starting such a newsletter. It is intended to promote communications between the Labex participants. This communication on the one hand consists of announcements from the secretariat regarding technical and operational matters. On the other hand this Newsletter will be a forum for the mutual exchange of information between participants. This means that the newsletter is open for contributions from all who are involved in soil analysis. When you encounter a problem in soil analysis, when you wonder whether others have met a similar problem and how they have solved it, you can bring it forward for publication in the Newsletter. The emphasis however will be on practical problems.

The first contribution to the newsletter comes from our Australian colleague Stan McLeod from CSIRO. He describes a simple home-made end-over-end shaker. In soil analysis extraction is of utmost importance and standardization of analyses starts with uniformity of shaking. This can be obtained by using this shaker.

We intend to publish the Newsletter whenever the need arises. This means that there will be no fixed time schedule with appearances will be at irregular intervals. The layout is not definitive but may also change from edition to edition. We hope that the Newsletter will live up to its expectations and that it may find its place in the soil analytical world.

From your secretary's desk

a) Programme Operations

During the last months two things absorbed the secretariat completely:

- in the first place the rounding-off of the activities in connection with the workshop last August,
- in the second place the securing of funds to extend the programme for another couple of years.

The rounding up of the workshop activities took considerably more time than anticipated. First, the discussions and recommendations were compiled and sent to all Labex participants and other interested parties. Next attention was given to the workshop proceedings. The contributions made by the various participants had to be edited, translated or partly rewritten. This was a time consuming affair but at the moment of writing this report, the text is being printed.

A project proposal was written in order to the secure funds for the next phase of the programme. A Dutch version was submitted to the Dutch Government Directorate of International Cooperation, and an English version has been sent to various donor agencies.

See the next paragraph for more news on this.

At the workshop it was recommended that before starting a new sample exchange round the analytical methods should be clearly written and sent around to the participants to clear ambiguities and avoid misunderstandings. Therefore we have started with the assistance of our Laboratory staff here at ISRIC, to draft these procedures. A number of them you will find enclosed with this Newsletter. As the other procedures become available they will be published with forthcoming Newsletters. If you feel that these procedure descriptions are unclear or ambiguous, please feel free to comment. Your remarks on the language, the wording, and on the analytical techniques are welcome.

b) Prospects on the programme extension

In December 1986 proposals for a programme extension were submitted to various donor agencies. This was followed in January 1987 by letters to our participants regarding their financial support of Labex. Until today we have received quite a number of supporting letters and commitments from participants to pay a fee for their participation.

Last month the German Agency for Technical Cooperation (GTZ) has committed itself to sponsor 5 participants, and the USAID Soil Management Support Services (SMSS, U.S.A.) also agreed to sponsor 5 participants.

The Dutch Government Directorate for Technical Cooperation (DGIS) has advised us that they will support Labex for the remainder of 1987. Support for the period thereafter is still under consideration by the various advisory bodies in DGIS.

All support is very welcome and although the total amount is not yet sufficient to ensure the extension of the programme over a prolonged period, it makes us confident that we can obtain the missing funds. We thank all who reacted to our request, as all responses help to convince donors of the importance of the programme.

Soil Sample Exchange 1987

The Soil Sample Exchange 1987 will be slightly bit different from the exchanges in 1986 and 1985. The changes follow the outcome of the workshop discussions last August.

The main difference will be in the mailing of samples. This time, instead of mailing the same set of samples to all participants, this time a list of available samples will be sent to you, with an order form, with which you can order the samples you wish to analyze. Samples will be mailed to you when we have received your order form.

The analytical procedures to be used are being distributed for comments prior to their use (see the relevant paragraph in this newsletter).

The provisional time table for the soil sample exchange 1987 is as follows:

distribution of procedures	
for comments	may/june
distribution of sample list	
with order form	june
mailing of samples	june/july
distribution of procedures	july/august
submitting of analytical results	february 1988

In this time table the interval between the mailing of samples and the submission of the results is approx. 6 months. As the mailing time of samples is usually longer than that of the paperwork the samples are sent first. Further details, also on the reporting of the results will follow in due time.

Draft Analytical Procedures

Complying with the workshop recommendations we will distribute the analytical procedures to all participants prior to their use in the Labex programme. Comments are invited and will be used to further improve the methods. This should ensure that in the next exchange round the analytical methods are at least comprehensible to all participants.

Please return your comments as soon as possible to us.

The draft procedures will be sent to you as they come available. To this newsletter the first are attached. To avoid confusion when newer versions supersede the old ones, the pages are numbered. E.g. L8703-24-1-1 means:

L8703: year and month of compilation

24 : code for type of analysis

1 : version no. 1

1 : page number of this procedure

This numbering should help you to keep your Labex-procedures-file organized.

Questions and Answers

Under this heading we will place questions that were posed to us, which we presume are also of importance to a wider audience. Name and address of the sender are given with the question. If you want to respond you can write directly to the person concerned. If you do, please send a copy to us, so we can possibly publish it in the next Newsletter.

Q: We are starting a bibliographical investigation to the quality of chemical soil analysis, to improve the quality of our analytical work. Can you send us information on this subject?

Ing. Norberto Bautista Aroche
Departamento de Suelos
Universidad Autonoma Chapingo
56230 Chapingo, EDO.
MEXICO.

A: The following books we can recommend:

- Massart, D.L., A.Dijkstra and L. Kaufman, 1978. Evaluation and Optimization of Laboratory Methods and Analytical Procedures. Elsevier Scientific Publishing Company, Amsterdam & New York. ISBN: 0-444-41743-5.

- Kateman G. and F.W. Pijpers, 1981. Quality Control in Analytical Chemistry. Wiley-Interscience, New York. ISBN: 0-471-46020-6. These books are not specifically directed at soil laboratories but cover statistical aspects of analytical chemistry in general.

Q: We are searching for the possibility to exchange the pipette method of soil particle size distribution for an instrumental method. Can you give us your opinion regarding adequate particle sizers?

Mrs. Nina Vajna

Plant Protection and Agrochemistry Centre

Ministry of Agriculture and Food

Pf.: 127

H-1502 Budapest

Hungary.

A: We ourselves have no experience with such instruments, nor have our colleagues in other soil laboratories in Wageningen. A colleague from the Physical Geography Department from Amsterdam University informed me that their experiences with particle size analysis by turbidity measurements were negative as it proved to be very difficult to obtain reproducible results with different soil types.

Article

A SIMPLE END OVER END BOX SHAKER

S. McLeod

CSIRO Division of Soils

Private Bag no. 2

Glen Osmond, South Australia 5064

Introduction

There are no critical measurements to this unit, you can either copy the example or have it made smaller or larger depending on the need. The box dimensions were determined by the 4 most convenient plastic bottle sizes that we commonly use and are locally readily available. Others should modify the boxes to suit their own containers. The combination of reduction gear and pulley sizes are similarly optional as long as the final shaker velocity is about 14 rpm (revolutions per minute). It is advisable to seal the wood with an epoxy paint, at least three coats, and to number the compartments. While in use the shaker must be balanced, i.e. the same number of bottles must be placed in opposite boxes and a blank containing water used when an odd number of samples is to be shaken.

Details

The frame work is constructed with angle-iron and is welded together as shown in the diagram. The motor is mounted at right angles to the axle thus an angle reduction drive is necessary. This makes the unit more compact however a simple reduction drive can be used with the motor mounted at right angles to that shown with the necessary adjustment to the length of the frame.

The motor is mounted on an angle-iron frame platform hinged on to the main frame so that the tension on the belt is the weight of the motor. This acts as a very effective clutch mechanism especially if the shaker is well out of balance. The motor used here is 0.25 hp, 240 V, 50 Hz, 1 phase. The reduction gearing (about 60:1) is connected to a 45 mm pulley which drives a 120 mm pulley on the box angle. This results in a shaking velocity of 14 rpm.

The box of the shaker comprises of 4 boxes each divided into 2 compartments as shown. The boxes have identical dimensions and are constructed with 12 mm marine ply for the sides, lids and divisions and 4 mm marine ply for the bases. Each compartment has the capacity to hold 48 * 100 ml (2 layers of 24), 15 * 250 ml, 12 * 500 ml, or 6 * 1000 ml round plastic bottles. The base of each compartment is covered with 25 mm soft plastic foam sheet. The 2 sets of 5 mm slots in each compartment take ply wood separators necessary when the whole area is not required. The slots are positioned to give the maximum number of combinations- from 1 - 4 rows of 100 ml, 1 - 3 rows of both 250 ml and 500 ml and 1 or 2 rows of 1000 ml bottles. The taller separators are for use with both the tall bottles and the 100 ml bottles when more than 24 are required (i.e. they are stacked). Eight separators of each size are sufficient and will cover all combinations. There are 4 sets of holes inside the front and rear walls of each compartment. These holes are strengthened with brass inserts to take the barrels of the fasteners and are for the 4 lid positions which together with soft foam packing easily accommodates any of the bottle sizes. For simplicity the lids are kept in place by 4 sliding bolts as shown and an ordinary wood knob screwed to each lid makes taking on and off more convenient (these had not been installed at the time of the photograph shown here). Modifications should therefore be made to box sizes, position of the separator slots and lid positions to fit ones own range of bottles.

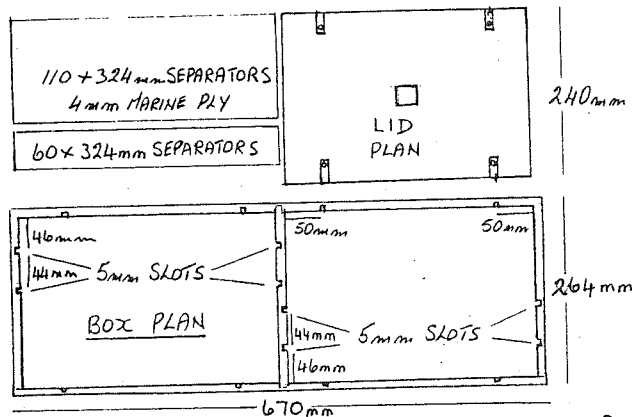
The shaker box is then put together as follows:

The base of one is glued to the side of the next etc. as shown to form one large box with a square hole running through its centre. NOTE- For those modifying the box sizes note that the boxes are not square, the sides are wider than the bases otherwise no hole would be left to take the axle shaft. Two flange plates are made from 5 mm steel with circular collars welded to take the shaft, are bolted to each end of the box with a counter-sink bolt through each small box as shown. The collars are drilled to take either a steel dowel or a heavy duty split pin. The two flange bearings for the axle shaft are bolted to the circular plates on the frame and the shaft passed through both the flange bearings and the box flanges thus suspending the box. The shaft is then

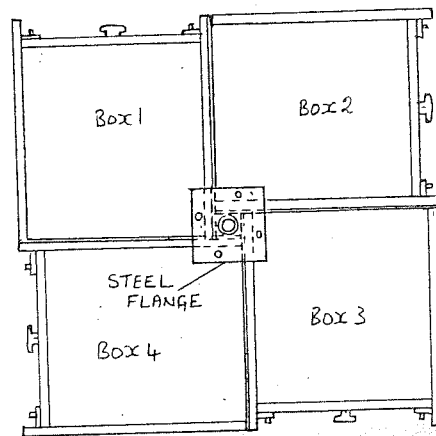
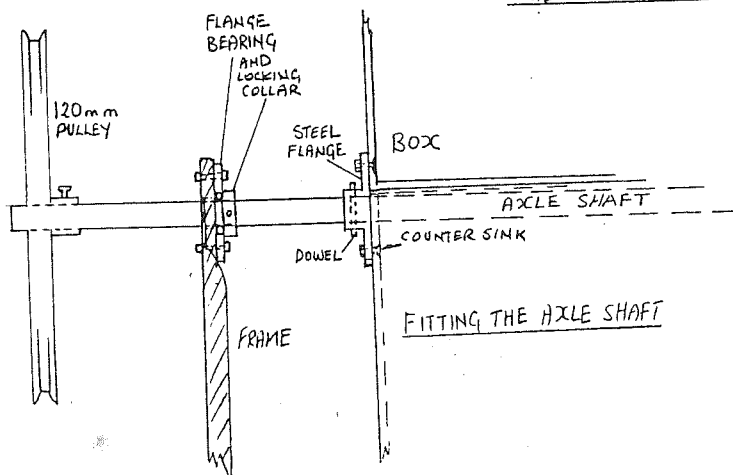
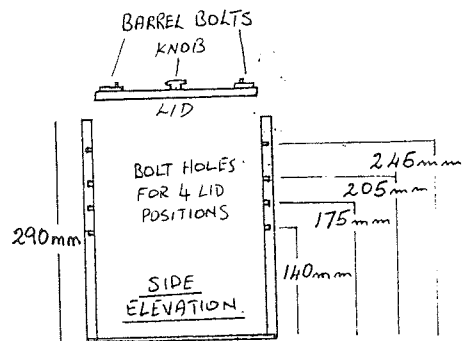
fixed in position using the locking collars which should be part of the flange bearings. The 120 mm pulley is then fitted on the end aligning it with the 45 mm pulley on the motor drive below. The box is then centred on the shaft, the holes drilled through and the dowels or split-pins fitted. An appropriate drive belt is easily fitted by raising the motor platform. We have attached a power switch to the front frame upright and the whole unit can either sit on the floor or, as we have it, on a standard laboratory bench.

Materials

Angle-iron main frame	30 * 5 mm	7 metres
Angle-iron motor frame	25 * 3 mm	1.5 metres
Steel hinge rod	200 * 12 mm diam.	steel rod
Flange plates	120 * 250 * 12 mm steel plate	to make two
Axle shaft	1 metre * 22 mm diam.	steel rod
Motor	.25 hp, 240 V, 50 Hz, 1 phase	
Gearing	angle reduction drive (60:1)	
Pullies	1 * 45 mm, 1 * 120 mm diam.	
Drive belt	Dunlop power V-belt A36-A960	
Axle bearings	2 * flange bearings	
Box sides, lids divisions	1800*900*22 mm marine ply	(3 sheets)
Box bases separators	1800*900*4 mm marine ply	(1 sheet)
Box flanges	100*200*5 mm steel plate	(to make 2) (+ 60*22 mm ID * 35 mm steel tube)
Lid handles	8 wood or plastic door knobs	
Lid fasteners	32 barrel bolts +	128 brass inserts



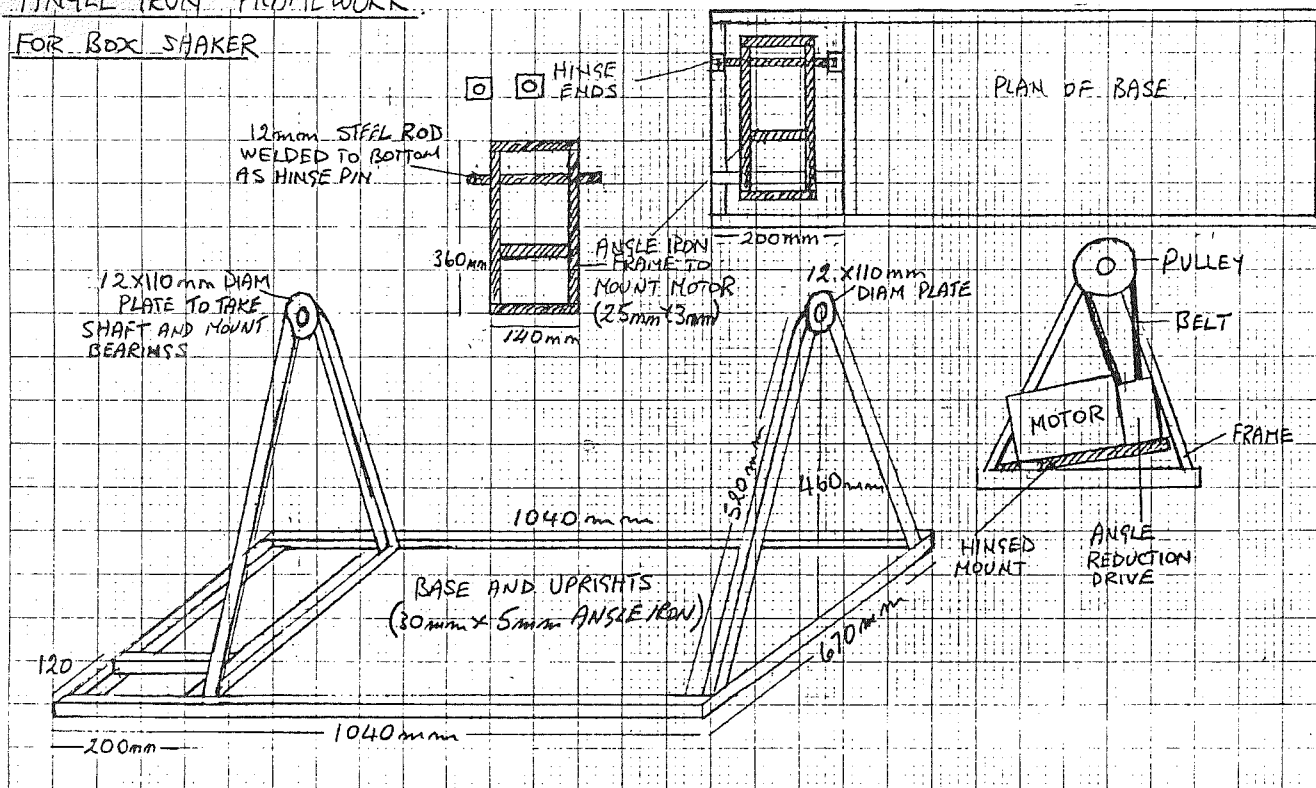
THE SHAKE BOX

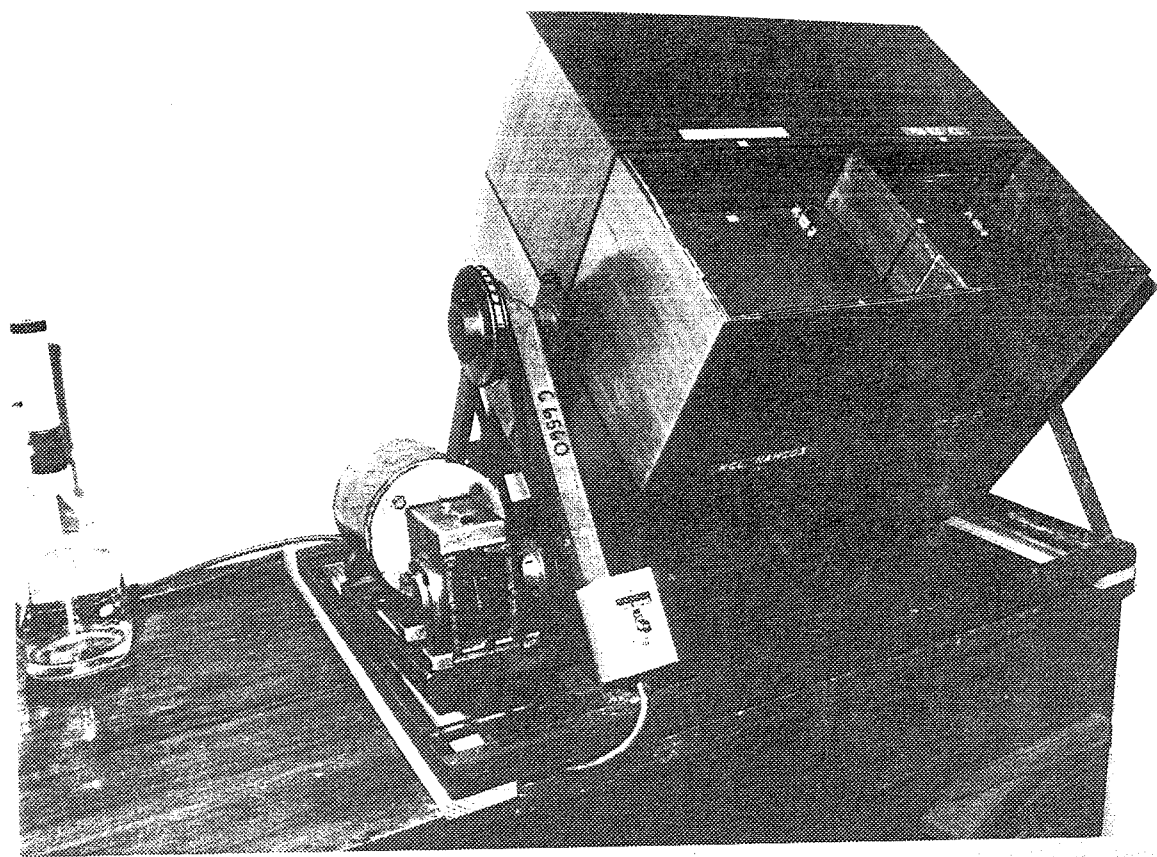


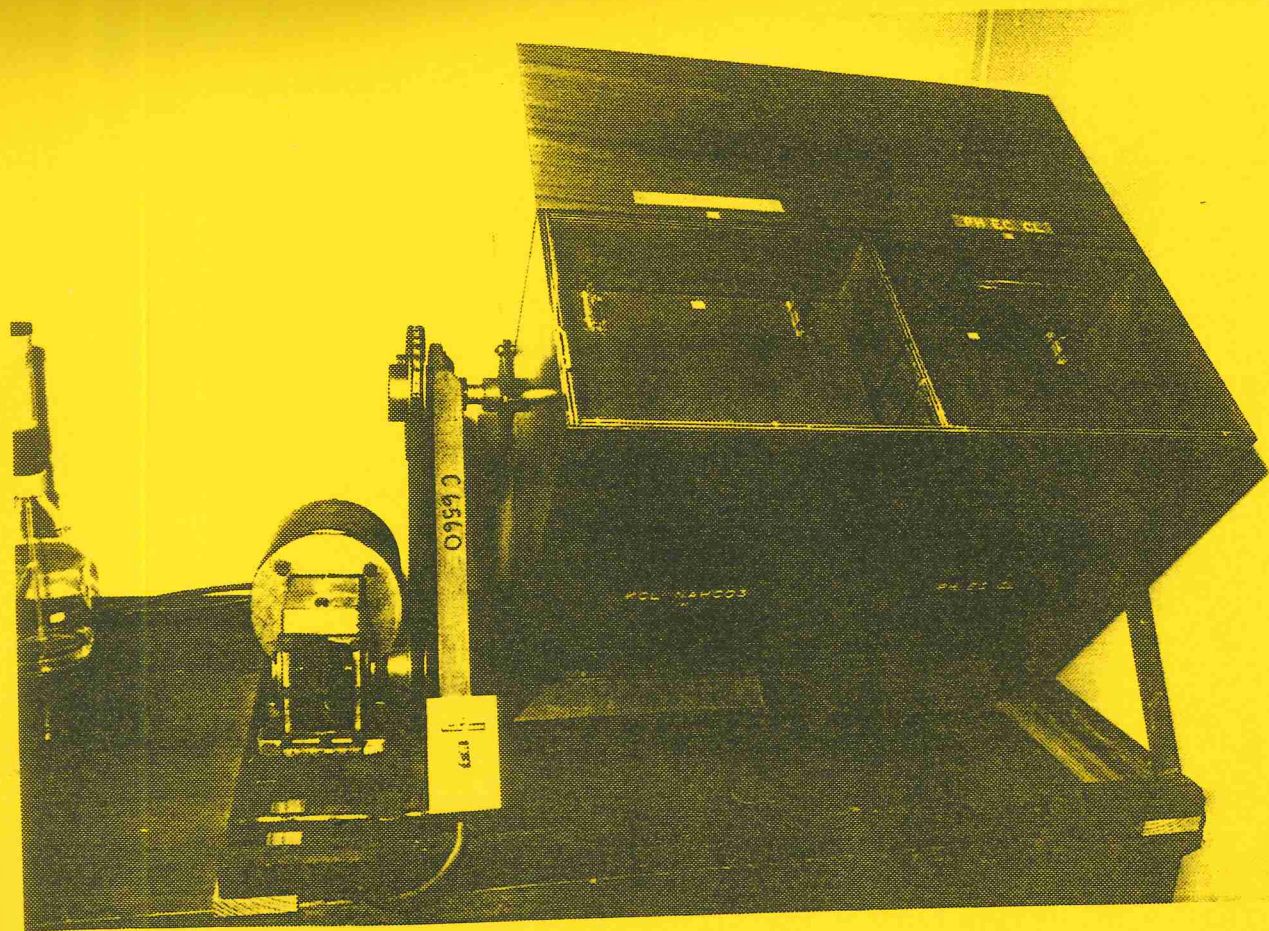
SIDE ELEVATION - THE SHAKER BOX

ANGLE IRON FRAMEWORK

FOR BOX SHAKER







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No. 2

September 1987

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(ce bulletin est également disponible en français)

From your secretary's desk

Since the previous edition of the Newsletter a number of things have happened. In the first place the proceedings of last years workshop came from the printer. One copy of this book is just one book, but 1100 copies means 30 packs stacked on 2 pallets! Anyway, after a couple of days everything was sorted out, put in the right envelopes and taken to the post office. Copies were sent to those who attended the workshop, to the other LABEX participants, and to institutes and libraries with which ISRIC exchanges publications.

Extra copies are still available for the modest price of 10 US dollars, including postage.

In the meantime I have already received an erratum from one of the authors. If you find more mistakes, please let me know.

On the first Newsletter I have got a reaction from Dr. Griepink of the Community Bureau of Reference (BCR) of the European Communities in Brussels. BCR has a number of soil and sewage sludge samples available, certified for the contents of Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Se and Zn. Though analysis of these elements is outside the scope of LABEX, this information might be of interest to you.

The section 'Questions and Answers' is not present in this issue by lack of proper topics.

In June your secretary attended a meeting of the International Standards Organization (ISO) of the Technical Committee on soil quality. This Technical Committee covers subjects as diverse as terminology, biological degradation and chemical analysis. This last topic is closely related with LABEX and a liaison is important. A report of this meeting you will find in this Newsletter.

Finally we are very busy these days with the preparation of the soil samples for the next exchange round. This means grinding, sieving and mixing of the dried material, which is a dusty affair. After that each sample is packed in small plastic bags and these bags have to be sealed. As the number of participants is growing the amount of work is also increasing. However be assured that this is not a complaint! On the contrary, a growing Labex programme is a recognition of our work, and this pleases us very much.

L.K. Pleijsier

Newsletter in French language/Bulletin en langue française

Ce Bulletin a été traduit en français par l'une de nos secrétaires, qui est une française. La traduction de ce Bulletin demande beaucoup de temps; pour cela nous souhaitons, pour les bulletins à venir, nous limiter à la traduction des messages les plus importants. Si vous avez reçu le texte anglais, mais que vous désiriez la version française, veuillez nous le faire savoir. En ce qui concerne la correspondance entre les participants de LABEX et le secrétariat, vous pouvez nous écrire en français, espagnol, portugais ou allemand, mais la réponse aux lettres par le secrétariat LABEX sera toujours en anglais.

Errata to LABEX Workshop Proceedings

Mr. Begheyn informed me on two errors in his article on CEC analysis with LithiumEDTA (page 46). In the first place the full address of his institute should read: Department of Soil Science and Geology, Agricultural University, Wageningen, The Netherlands.

The second erratum is more serious: On page 47 the second equation should be:

$$K = (MY) / (M).(Y)$$

This is the inverse of what is mentioned in the proceedings. On page 49 after the first equation is written "(in extract)". This should be "(mole ratio in extract)".

The remark about the address naturally also applies to his article on Organic Carbon analysis on page 124.

Soil Sample Exchange 1987

Timetable:

As you will have noticed the timetable for the Soil Sample Exchange 1987, as given in the previous Newsletter was very optimistic, and inevitably needs modification.

As it looks now this Newsletter no. 2 will be mailed in September 1987. With it the order form of the samples is distributed.

The samples will be mailed to you after we have received your order form, so this will probably be around October.

Results will have to be returned to us approximately 1/2 year later, so in the first quarter of 1988. The exact date before which the results will have to be reported, will be announced later.

Samples:

For the Exchange 1987 we have made available 10 samples. Some of them are brand-new, others have been employed earlier in one of the previous exchanges. In the listing on the separate sheet some of the analytical data are summarized. These data are only rough indications of the soil parameters and are meant to give you an idea of the nature of the soils. Of course it is perfectly well possible that your results will deviate from those in the list.

The lower part of the sample list is an order form. Please tick on this form the samples you wish to analyze, and return it to us. Do not forget to enter your name and/or institute and

country. Only after we have received your form we will mail the samples. The mailing address will be similar as on the label used for this Newsletter. In case this needs correction please return the corrected label also to us.

We would appreciate to have the forms not later than 31 October 1987.

The soil samples will be sterilized in Wageningen by radiation with Gamma-rays, prior to mailing. A certificate of this treatment will be sent to you to help you with the importation, at your customs and/or quarantine offices.

Soil parameters to be analyzed:

The soil parameters to be analyzed are those for which the procedures are provided. They are listed on the sheet accompanying the procedures.

Analytical procedures:

To improve comparability of the data the LABEX analytical procedures are to be used. In case you have to deviate from this rule, because of lack of the proper apparatus, glassware or chemicals, you may do so, provided you indicate this on the reporting form. In that case your data will be treated separately.

Some participants may have the opinion that their own routinely used procedures produce better figures, and therefore they prefer using those. However it should be pointed out that the aim of LABEX is to reduce the variability in the analytical data by standardization of methods. This will allow mutual comparison of data between participants. Only those participants, who do not have the possibility to follow the LABEX procedures and who would otherwise not submit data, may deviate from the LABEX analytical procedures.

Reporting of results:

When reporting the data at least duplicate results are necessary. This was discussed at last years workshop. These duplicates should be 'real duplicates'. This means that the entire analysis, from subsampling to meter reading, should be duplicated and not only the final step with the reading.

The form in which you have to enter your figures will follow in due time and will be sent with one of the next Newsletters.

Draft Analytical Procedures

A number of participants already commented on the procedures sent last June. Comments will be used to revise and further improve the methods. This should ensure that in the next exchange round the analytical methods are at least comprehensible to all participants. Revised versions will be sent with one of the next newsletters.

With this Newsletter again a number of procedures are sent. Comments are still welcome. Please return your remarks as soon as possible to us.

Regarding the alternative procedures for the CEC, it might be interesting for you to know that the choline chloride method, the cobalti hexamine method, the lithium barium EDTA method and the silver thio urea method are currently being tested by a small group of LABEX participants. As these methods are relatively new

and uncommon, testing by a small group prior to their use in LABEX, was recommended in the workshop discussions. We will inform you of course on the results of this testing as they become available.

ISO Technical Committee on Soil Quality
Second meeting in Milan (Italy), June 1987

What is an ISO Technical Committee and why is it initiated?

In 1986 the International Standards Organization (ISO) established a Technical Committee (TC) on soil quality. The goal of this TC is to standardize terminology, analyses (chemical, physical and biological) and sampling methods, all related to the field of soil quality. This initiative stems from the need of environmentalists for international rules and regulations regarding soil pollution. International regulations require international comparability of analytical data, obtained with standardized procedures. The soil pollution aspect is reflected in the great attention given to work items as heavy metals, pesticides, hydrocarbons etc. An ISO technical committee will not set limits of acceptability of contaminants. Only terminology and methodology will be standardized.

Who take part in the ISO Technical Committee work?

The member bodies of ISO are the national standardization institutes, as DIN for Germany Federal Republic, BSI for United Kingdom or ANSI for the USA. Member bodies can express their willingness to participate in a technical committee. At present there are approx. 200 technical committees. The committee on soil quality is TC 190. In TC 190, 11 countries participate as P-member (participating member) and 18 as O-member (observing member). P-members are: Austria, Belgium, France, Germany F.R., Hungary, India, Italy, Jamaica, Kenya, Netherlands, and United Kingdom. O-members are: Australia, Canada, Chile, China, Czechoslovakia, Denmark, Egypt Arab Rep., Greece, Malaysia, Mexico, Rumania, South Africa, Spain, Sweden, Switzerland, Turkey, USA, and Yugoslavia. Generally the member bodies rely for specific knowhow of the work items on experts from national universities or research institutes.

The International Society of Soil Science (ISSS) has an official liaison with this TC. This means that ISSS is kept informed on the developments.

At the TC meeting in Milan, June 1987, the following countries were represented: France, Germany F.R., Hungary, Italy, Netherlands, Sweden, United Kingdom. From the liaison organizations only ISSS had sent a representative, Ir. L.K. Pleijssier (on behalf of Dr. W.G. Sombroek, ISSS Secretary-General).

Work items of TC 190

At the second TC 190 meeting in June 1987 in Milan, the work items were endorsed and allocated to subcommittees. Secretariats of subcommittees were assigned and convenors of working groups were designated. These convenors will have to be the driving force behind the work of the working group.

The subcommittees are:

TC/ Subcommittee:	secretariat:	work item:
TC 190-SC 1	AFNOR(France)	terminology and codification
TC 190-SC 2	DIN(FRG)	sampling
TC 190-SC 3	DIN(FRG)	chemical methods and soil characteristics
TC 190-SC 4	BSI(UK)	biological methods
TC 190-SC 5	NNI(NL)	physical methods

The work items of subcommittee 3 have common ground with LABEX. This subcommittee has the following working groups and convenors:

WG:	convenor:	work item:
SC 3/WG 1	Dr. W. Bitter (FRG)	heavy metals, As, Se
WG 2	Dr. W. Huber (FRG)	nitrogen compounds
WG 3	?	sulphur compounds
WG 4	Mrs. Dr. A. Horvath (H)	cyanide
WG 5	Dr. A. Barbera (I)	phosphor compounds
WG 6	Dr. K.G. Liphard (FRG)	mineral oil
WG 7	Dr. W. Ebinger (FRG)	chlorinated pesticides, PCB's, chlorinated hydrocarbons
WG 8	Dr. V. Houba (NL)	electrical conductivity, pH, redox potential, CEC
WG 9	?	particle size analysis

Working groups 5, 8 and 9 are important for us. During subcommittee and working group discussions efforts were made to identify the pitfalls on the way to standardization.

What is the relevance of this TC for LABEX?

In the coming period the convenors of working groups will start drafting proposals for standards. Therefore national standards or related documents are invited. The LABEX analytical procedures will also be brought to the attention of the working groups. It will probably take a number of years before international standards will be published. By that time the LABEX procedures may need modification in order to comply as much as possible with these standards.

However, from the list of participating member bodies it appears that they are predominantly from European countries, where soil pollution is also a political issue. Therefore it is thinkable that the ISO standards will be suitable for highly sophisticated laboratories only, and limited to soils in the moderate, 'European type', climate. In that case LABEX procedures may deviate from the ISO standards, to cater for a wider range of soils and to be applicable in laboratories without resource to expensive equipment.

In any way we will follow closely the developments in this TC. As

news regarding the relevant working groups becomes available we will inform you. A more general report of this TC 190 meeting, also about the other subcommittees, will be published in the ISSS Bulletin later this year.

BCR Certified Reference Materials for Trace Element Soil Analysis

B. Griepink
Commission of the European Communities
Community Bureau of Reference
rue de la Loi 200, B-1049 Brussels, Belgium
(phone: +32-2-235.88.12)

Analytical quality control requires, besides a good control within the laboratory, also participation in inter-comparisons and, wherever available, the use of certified reference materials. In view of the disagreement of analytical results often encountered in soil analysis, the Community Bureau of Reference (BCR) has produced and certified three soils: a calcareous loam soil (BCR CRM 141), a light sandy soil (BCR CRM 142) and a sewage sludge amended soil (BCR CRM 143). In addition the BCR has produced three sewage sludge reference materials.

All these materials have been certified for total trace element contents (e.g. Cd, Hg, Pb, Zn, Ni ...). The table below gives a list of elements and certified contents.

The preparation, homogeneity and stability studies as well as the certification analyses are given in detail in a certification report which is delivered with the soil sample.

Two publications cover the subject additionally,
on soils:

- B. Griepink, H. Muntau, H. Gonska, E. Colinet:
Fres.Z.Anal.Chem. (1984) 318:588-591

on sludge:

- B. Griepink, H. Muntau, E. Colinet:
Fres.Z.Anal.Chem. (1984) 318:490-494

BCR certified samples

SOILS:

CRM 141
calcareous loamCRM 142
light sandCRM 142
sewage sludge
amended soil

	microgram/gram	microgram/gram	microgram/gram
Cd	0.36 \pm 0.10	0.25 \pm 0.09	31.1 \pm 1.2
Co	(9.2)	(7.9)	(11.8)
Cr	(75.0)	(74.9)	(228)
Cu	32.6 \pm 1.4	27.5 \pm 0.6	236.5 \pm 8.2
Hg	0.0568 \pm 0.0043	0.104 \pm 0.012	3.92 \pm 0.23
Mn	(547)	(569)	(999)
Ni	(30.9)	29.2 \pm 2.5	99.5 \pm 5.5
Pb	29.4 \pm 2.6	37.8 \pm 1.9	1333 \pm 39
Se	(0.16)	(0.53)	(0.6)
Zn	81.3 \pm 3.7	92.4 \pm 4.4	1272 \pm 30

Aqua regia
soluble:

Cd	(0.30)	(0.22)	(31.5)
Cr	(53)	(44.4)	(208)
Cu	(31.2)	(25.3)	(236)
Mn	(512)	(527)	(935)
Ni	(28)	(28.9)	(92.7)
Pb	(26.3)	(30.9)	(1317)
Zn	(70)	(80)	(1301)

Matrix	milligram/gram	milligram/gram	milligram/gram
SiO ₂	(425.8)	(682.2)	(427.2)
CaO	(179.8)	(49.4)	(93.5)
MgO	(11.9)	(10.9)	(49.0)
Al ₂ O ₃	(105.6)	(94.8)	(101.3)
TiO ₂	(4.7)	(6.2)	(6.7)
Fe ₂ O ₃	(37.4)	(28.0)	(37.5)
P ₂ O ₅	(1.6)	(2.2)	(9.1)
Na ₂ O	(4.3)	(9.7)	(4.1)
K ₂ O	(15.6)	(24.1)	(16.2)

Loss at 900 °C	0.2065	0.0853	0.2434
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SLUDGE:

	CRM 144 sewage sludge domestic origin	CRM 145 sewage sludge	CRM 146 sewage sludge industr. origin
	microgram/gram	microgram/gram	microgram/gram
As	(6.7)		77.7 \pm 2.6
Cd	4.82 \pm 0.97	18.0 \pm 1.2	11.8 \pm 0.7
Co	9.06 \pm 0.60	8.38 \pm 0.71	(784)
Cr	(485.4)	(105.4)	934 \pm 24
Cu	713 \pm 26	429 \pm 10	9.49 \pm 0.76
Hg	1.49 \pm 0.22	8.82 \pm 0.88	588 \pm 24
Mn	449 \pm 13	241 \pm 12	280 \pm 18
Ni	942 \pm 22	41.4 \pm 2.4	1270 \pm 28
Pb	495 \pm 19	394 \pm 15	(1.7)
Se	(2.3)	(3.3)	4059 \pm 90
Zn	3143 \pm 103	2843 \pm 64	
Aqua regia soluble:			
Cd	(3.6)	(16.8)	(76.5)
Cr	(494)	(85.2)	(769)
Co	(8.6)	(6.8)	(9.9)
Cu	(694)	(415.9)	(921)
Mn	(436)	(219.6)	(555)
Ni	(947)	(38.5)	(269)
Pb	(479)	(332)	(1255)
Zn	(3090)	(2772)	(4002)
Matrix	milligram/gram	milligram/gram	milligram/gram
SiO ₂	(136.4)	(217.0)	(228.0)
CaO	(56.8)	(153.5)	(142.0)
MgO	(9.2)	(31.0)	(33.0)
Al ₂ O ₃	(45.8)	(34.4)	(90.0)
TiO ₂	(1.9)	(3.2)	(29.1)
Fe ₂ O ₃	(63.4)	(13.0)	(26.5)
P ₂ O ₅	(50.8)	(75.3)	(59.0)
Na ₂ O	(4.6)	(2.7)	(3.1)
K ₂ O	(7.8)	(4.9)	(5.8)
Loss at 900 °C	0.620	0.451	0.377

Values in brackets are not certified.

The certification report describes in detail the analytical procedure to obtain the aqua regia soluble content of the elements.

The samples are in the form of powder in bottles containing 40 gram approximately.

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LABEX NEWSLETTER

No. 3

November 1987

Labex secretariat:
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6700 AJ Wageningen
The Netherlands.

From your secretary's desk

This third issue of the Newsletter will be devoted mostly to the next soil sample exchange. There are a number of organizational topics that need to be clarified. Moreover a number of more theoretical points have arisen from the correspondence with the participants.

Regarding the analytical procedures we received also a number of comments. We have tried to answer them satisfactorily. However some question marks cannot be solved instantly but need further research. Possibly some participants have unpublished data of internal lab research on these topics. Even when such data are not suitable for publication in an official, refereed journal, it might be interesting for the other LABEX participants to be informed on the outcome of such research via this Newsletter.

The LABEX programme's aim is in the first place to serve the participants. Therefore if there is anything you would like to ask or to comment upon, do not hesitate to write to me. When the subject might be of interest to the other participants as well, I will give the answer to you via the Newsletter.

It is there for you.

L.K. Pleijsier

Soil Sample Exchange 1987

Timetable:

The mailing of the samples started in the first week of November, but only to those participants whose order form we have received. It is still possible to send in the order form. In that case the samples will be mailed to you upon reception of your form.

The deadline for submitting data will be May 1st 1988. This should be a sufficiently long period. It will enable participants to finalize the analyses well in time.

A summary report with first results is planned shortly afterwards.

Samples:

The available soil samples in LABEX exchange 1987 are listed below:

<u>no.</u>	<u>soil type</u>	<u>Horizon</u>	<u>depth</u>
38	Calcaric Cambisol	A	0-18
39	Calcaric Cambisol	AB	35-60
40	Orthic Acrisol	E	10-35
41	Orthic Acrisol	EB	35-60
42	Pellic Vertisol	?	?
43	Orthic/Humic Podzol	Bs	?
44	Gypsic Yermosol	C	40-50
45	Xanthic Ferralsol	Ah	0-15
46	Xanthic Ferralsol	Bt	80-150
47	Pellic Vertisol	A	0-20

Soil parameters to be analyzed:

The soil parameters to be analyzed are those listed on the data sheet. This data sheet is enclosed with this Newsletter. For these parameters the analytical procedures were sent with previous Newsletters, except for CEC and exchangeable cations. These are sent herewith. This list of procedures is a very long one and you may not be able to carry out all. You are however completely free to do all or only part of these analyses. For instance if you normally use the hydrometer method for texture and you have no possibility to do the pipette method, you may send in only the results you obtain with the hydrometer.

The carbonate determination obviously only makes sense when the sample has shown to contain carbonate.

So you are free to choose both from the soil samples and the methods, those that suit you most. However the programme gains in value as more data are sent in.

Analytical procedures:

The LABEX analytical procedures to be used are those that were sent with Newsletters # 1 and 2. In addition a CEC procedure is sent herewith. The procedures were initially drafts on which comments were invited. Received comments are discussed below in the separate paragraphs. These have lead in some cases to modifications. Please take good notice of these amendments as no modified procedure description will be sent now.

Regarding the CEC, see also the separate paragraph.

Note: In the previous Newsletters it may not have been announced clearly enough that in addition to the exercise with the LABEX procedures participants are invited to carry out the analyses also with their own routine methods, and submit results. This will permit comparison of participants routine work with others'. Please indicate this clearly on your data sheet and make sure to send us your detailed procedures, if you have not done so in the past.

Comparability of data is increased by standardization of methods. As international standards are still under development we have drafted the Labex procedures. A special paragraph in this Newsletter is devoted to this topic of 'Standardization versus Flexibility'.

To obtain a valid comparison of results, the data from the Labex procedures will be tabled separately from the data obtained with other methods.

Reporting of results:

For the reporting of results the enclosed data sheet is provided. This sheet is quite large to provide ample room for your data. The form is laid out as a table. In the uppermost line you read the sample numbers. In the next line you see 'I' and 'II' under each sample number. These numbers refer to the replicates. As stated before you are kindly requested to provide replicate results.

After each analysis the unit, in which the result is to be reported, is stated.

In the column under 'code' you find a two digit number followed by a single digit number. These numbers refer to the analytical procedures.

The two digit number corresponds with the code as printed on the top left of every page of the procedure description.

The one digit number is added to provide for procedures giving more than one result.

For instance: dithionite extraction is coded on the data sheet as '61 1', '61 2' and '61 3'. The analytical procedure is numbered L8703-61. This procedure results in data for Fe, Al and Mn, which are to be entered on the appropriate lines.

When you employ an analytical procedure that deviates considerably from the LABEX method, you should clearly cross out the number in the code column or use additional paper (e.g. photocopy of the data sheet).

Comments on the Draft Analytical Procedures

A number of participants commented on the draft analytical procedures that were sent with the Newsletters no. 1 and 2. These remarks and suggestions are given below with our commentaries, grouped per analysis. We are very grateful to those who took the trouble to comment.

pH

- Under Reagents it is erroneously mentioned that the Calcium-Chloride solution to be used is 0.02 M. This should be 0.01 M, which is in accordance with the 1.47 g/l.
- We have proposed a shaking time of 2 hours, immediately followed by measurement with the electrode in the upper part of the suspension. Some participants prefer a short shaking time followed by a long period during which the suspension can settle. A short shaking period has for calcareous soils the disadvantage of possibly not reaching an equilibrium between the suspension and the carbonates. Therefore we prefer the 2 hours shaking.
- It has been remarked that KCl flowing from the reference- or combination electrode may influence the EC when measured subsequently. It is our experience that the rate of KCl flow depends very much on the make and type of electrode. We did not find an appreciable effect on EC, but we are interested to learn experiences from other participants as well. To prevent this problem EC may be measured before pH assessment.

Amendment on page L8703-01-1-1: Third line under Reagents: 0.02 M should be 0.01 M.

Particle size analysis

- The pretreatments should be applied in the following order: 1) oxidation of organic matter, 2) removal of carbonates, 3) deferration. It would be welcomed if some participants would have the opportunity to compare results obtained with the sequence of pretreatment (1) _ (2) with data obtained with the sequence (2) _ (1). Comparison in ISRIC's laboratory showed no significant differences. Oxidation of organic matter is required for all soils. Removal of carbonates is only necessary when $\text{pH-H}_2\text{O} > 6.8$. Deferration is only applied in exceptional cases for specific research. So we should agree that for the LABEX samples deferration is NOT needed. Moreover, recent research in our ISRIC laboratory indicates that clay contents often shift only marginally after deferration. Possibly this treatment not only releases clay but may also remove comparable quantities of iron oxides from the clay fraction (Pers. Com. L.P. van Reeuijk). For the hydrometer method removal of organic matter and carbonates is identical to the pipette method.
- To oxidize the organic matter H_2O_2 is added. Initially 15 ml is added. The next day more H_2O_2 is added in small increments (30 ml) about every 2 hours. The oxidation is complete when the supernatant is clear. Usually this takes 1 or 2 days, but very humic soils may require more time (and more H_2O_2).
- To remove carbonates the acetate buffer $\text{pH}=5$ is added. The carbonate reacts with the acetic acid in the buffer. In case of very high carbonate contents a large quantity of buffer is needed. To supplement the acid neutralized by the carbonates, glacial acetic acid may be added. However care should be taken that the pH does not drop much below 5.
- At the LABEX workshop it was recommended that as shaking method the end-over-end shaker should be adopted. The recommended turning rate is 14 rpm. (see Stan McLeod's paper in Labex Newsletter no. 1). In case you do not have such or a comparable apparatus you can use a reciprocating shaker at a speed of approx. 125 strokes/min.
- As shaking time 16 hours is indicated, because this can conveniently be done overnight. Some laboratories prefer not to leave shakers on during the night. It seems that a shorter shaking time of 8 hours gives comparable results. Both the shaking method and shaking time may influence results. Possibly some participants have data on this.
- The plunger described at the hydrometer method, can also be used for the pipette method. This avoids the cumbersome shaking of the cylinders. However, care should be taken that the plunging action brings all soil particles in suspension. For some soils, in particular very silty soils, this might prove quite difficult.
- At the washing stages in the various pretreatment the soil material is allowed to settle in the beaker, the clear supernatant is siphoned off, water is added and the material is re-dispersed with a rubber policeman, etc. Alternatively the settling may be substituted with centrifuging in large centrifuge bottles.

- The depth of pipetting as given in table 1 is defined as the distance between the suspension surface in the cylinder and the tip of the pipette.
- Evaporating the pipetted suspension in the moisture tin should preferably be done on a water or sand bath, prior to drying at 105 °C. Evaporating suspensions directly in an oven might ruin this apparatus.
- In the calculations a correction for dispersing agent is used (1.000 g). This correction is based on the addition of 20 ml dispersing agent (50 g/l) to a final volume of 1000 ml. So the cylinder contains $50 \times 20/1000 = 1.000$ g dispersing salts. The correction can also be determined with a blank. This boils down to determining the residue after drying an aliquot of a fifty-fold dilution of the dispersing agent. Possible inaccuracy in the preparation of the dispersing agent is compensated in this way.

Amendment on page L8703-11-1-4: line 11. Delete: 'and the correction for dispersing agent becomes 1.250 g.'

- The textural fractions are expressed as percentages of the calculated sample weight. This sample weight is computed as the sum of the fractions (see page L8703-11-1-4). This means that removed organic matter and carbonates are not included in this calculation base.

Water dispersable clay

- The method for water dispersable clay comes down to a determination of clay without subjecting the soil to the pretreatment and dispersion, used in the 'conventional' texture method. The parameter determined with this procedure, is also completely different from other texture parameters. In erosion studies the water dispersable clay is used as indicator to assess susceptibility of the soil to erosion. In LABEX this parameter serves an additional purpose. As we expect that much of the variability in the texture data in LABEX originates from variabilities in the pretreatment, the variability in the water dispersable clay data is expected to be less.

Water Retention at 15 bar

- Special care should be given to the initial saturation of the samples with water. An alternative way to achieve this is to add water to the surface of the ceramic plate after rings and samples have been installed, and allowing the samples to equilibrate with the free water surface overnight.

Exchangeable Acidity

- The Sodium hydroxide solution 0.025 M is prepared by dissolving 1 g NaOH pellets in 1 l water, and NOT 4 g as is mistakenly indicated.

Amendment on page L8703-26-1-1: Reagents, subheading Sodium hydroxide solution, approx. 0.025 M. The sentence should read: 'Dissolve approx. 1 g NaOH pellets in water in a 1 l flask.'

Extractable Acidity

- In this procedure the soil sample is extracted with a buffer (BaCl₂-TEA) solution at pH=8.2. The extracted acidity is determined by titrating the residual buffer with HCl. The blank will use more HCl than the sample, and the calculation should be

based on the difference between quantities used for blank and sample. Amendment on page L8703-27-1-1: Calculation should be: 'Extractable Acidity (me/100 g soil) = $(b-a) \cdot 25 \cdot \text{mcf/s}$ '

Organic Carbon

- The Walkley & Black procedure involves an oxidation of the organic carbon with excess oxidant. This excess is backtitrated with Ferrous Sulphate. When the soil contains much organic material the oxidant excess might not be sufficient. Therefore if less than 2 ml Ferrous Sulphate (1M) is needed in the titration, the determination should be repeated with less soil.

Amendment on page L8703-31-1-1: In the paragraph procedure the last sentence above the note should read: 'If less than 2 ml ferrous sulphate solution is needed ...etc.'

- In the calculation on page L8703-31-1-2 a factor 0.39 is used. This figure incorporates the recovery factor (=1.3) for incomplete organic matter combustion. If you report your results using a different recovery factor, you should clearly indicate this with the data.

Carbonate

- 100 ml 1 M HCl is added to 5.00 g sample in a plastic bottle. The resulting CO₂ is allowed to escape. The next day the bottle is closed and shaken for two hours to complete the carbonate dissolution. To prevent this last bit of CO₂ 'to blow up' the shaking bottle, a slight under-pressure is applied by slightly indenting this bottle by hand, before tightening the cap.

Phosphorus Bray-1

- Under the reagents the formula for Ammonium Molybdate is incorrect. This should be: $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$
- The formula for Potassium Antimony Tartrate should be: $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$. This reagent is also used in the Olsen and Dabin procedures and the formula should be corrected there as well.

CEC

Regarding the alternative procedures for the CEC, we mentioned in the previous Newsletter that the new alternative CEC methods were being tested by a small number of LABEX participants. So far we have received analytical data from three of them.

Besides producing analytical results these participants have also heavily commented on the procedures.

These comments have convinced us that the descriptions of the procedures as they are now, are not suitable for distribution and testing on a wider scale.

So for the time being we will limit ourselves to the Ammonium Acetate method. The description of this method is enclosed.

To remind you, the other methods under consideration are:

- Choline Chloride method
- Cobalti Hexamine method
- Lithium Barium EDTA method
- Silver Thio Urea method

We will study the results and comments received from the selected participants. Findings will be publicized in one of the coming Newsletters. It is very well possible that descriptions of the alternative CEC methods will be distributed later, for use with the current samples no. 38 to 47. So we kindly ask you to retain the leftovers of Sample Exchange 1987, for future use.

Standardization versus Flexibility

A point of discussion, recurring every now and again, is the topic of standardization versus flexibility. In LABEX this comes up when participants do analyses using own procedures, instead of using the LABEX methods. Participants' own procedures often are a result of in-house research, with adaptations of procedures from the literature to local conditions, to the level of skill of the personnel, to availability of equipment and of chemicals. Understandably one is not inclined to discard all these efforts and adopt new methods because of one's participation in a programme such as LABEX. Moreover a good researcher will always be on the outlook for improvements in the laboratory methodology. This seems incompatible with our pursuit of standardization.

Standardization is a tool to promote interchangeability of products, services and information from one place on our globe to the other. We are using standards every day even without being aware of them. A much used example is the nut and bolt. A nut made in one factory should fit a bolt made elsewhere. And as long as all manufacturers of nuts and bolts adhere to the standard, the users do not even realize that standardization is involved.

In the standardization of analytical methods the same principles are involved, but they work out differently. In the example of nut and bolt disregard of standards becomes visible instantly, as nut and bolt do not fit. By contrast, disregard of standard methods in analytical methods can remain hidden for a long time. The result of a soil analysis is a numeric value and more often than not this figure is given without a clear explanation of how this result was obtained. Interpretation of the result or comparison with other data can go on unpunished for some time before the incompatibility of the data comes to daylight. At that moment very often the additional information on analytical methods or experimental conditions is irretrievably lost, which means the data have only limited value.

Standard methods are a help to validate comparison of data manufactured at different places or at a different time. To compare data obtained with own procedures, one should know how these procedures correlate with the standard methods. Moreover use of standard methods in an exchange like LABEX can disclose systematic errors that have long gone unnoticed. Therefore in the LABEX soil exchange we encourage participants to use the provided LABEX methods next to own methods.

It should be clear that LABEX does not discourage participants own research on new or modified analytical procedures. It will be of help in assessing the compatibility of these newly developed methods to existing practices.

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1988



NEWSLETTER

NEWSLETTER 4

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1 LABEX News

On June 1th 1988 Mr. L.K. Pleijsier, the secretary of the LABEX programme, was succeeded by Mr. J. Gerits. After serving a 3-year contract Leendert Pleijsier had to return to his original post. We would like to acknowledge and thank Leendert for his most valuable contribution to the LABEX programme. During the period from January till June 1988, most of the current LABEX affairs were handled by Dr. L.P. van Reeuwijk.

Recently Mr. L.K. Pleijsier was invited to give a presentation on the variability of soil analytical data from the LABEX programme at the international Symposium on "Land Qualities in Space and Time" (Wageningen, August 22-26, 1988). This presentation entitled "Variability in Soil Data", will appear in the Proceedings of the Symposium. If and when available we shall try to forward a copy to all LABEX participants.

A similar presentation was given by the present LABEX programme secretary, invited by IBSRAM, at the African Workshop for Acid Tropical Soil Management and Land Development Practices in Peru (Yurimaguas, August 28-September 11, 1988). This paper will be published by IBSRAM in the Workshop Proceedings. Finally 2 other presentations are planned for the IBSRAM Workshop on Site Selection, Characterization and Monitoring for Soil Management Networks in Thailand (Chiang Mai and Chiang Rai, November 7-18, 1988).

2 Soil Sample Exchange 1987

2.1 Progress

Due to the change of personnel, the deadline for submitting the analytical results of round 87 had been extended to August 15, 1988. Up to now, not all the participants have submitted their results. It should be kept in mind that only those participants who have submitted their results, will receive an Interim Report with all the results of Round 87. This Interim Report will appear in December 1988.

2.2 Logistics

Some participants reported not to have received the ordered LABEX soil samples. In order to avoid such mailing problems in the future, each participant is kindly requested to check whether the present mailing address is logistically attainable or should be replaced by a more reliable mailing route. Obviously there is a fair chance that samples will not arrive at their destination if it concerns mailing addresses in remote areas. In such cases it is recommended to make arrangements to use a more reliable mailing address; for example the main department in the capital.

Repeatedly participants ask for copies of the LABEX soil analytical procedures and data sheets to report their results. Apparently this LABEX documentation becomes easily "lost" in drawers and cupboards, especially after changes in personnel. We therefore suggest to prepare a duplicate file of all the LABEX documentation for daily use. The original file should be stored in a central place, inaccessible for common use.

It may be clear that the above mentioned problems cause a considerable delay in the progress of the LABEX programme. Therefore the participants are entreated to check at the beginning of each exchange round if their LABEX documentation is complete (data sheets and LABEX analytical procedures). They are also kindly requested to confirm as soon as possible the receipt of the LABEX soil samples.

For some participants the amount of sample was not enough to perform the analyses in duplicate according to the LABEX and their own procedures. In the next exchange round this problem will be avoided by prescribing a limited number of different analyses (ref. section 5).

2.3 The LABEX analytical procedures

From several participants we received most valuable comments on the prescribed LABEX analytical procedures. These comments are summarized below.

CEC (8703-21-1)

- For the distillation of adsorbed ammonium 1 N NaOH is used. This has replaced 35% NaOH, which should be omitted from the procedure.
- Generally 100 ml ethanol (96%) is sufficient to remove the excess of ammonium. Additionally Nessler's reagent can be used to check the efficiency of the washing. Alternatively a $\text{NH}_4\text{Ac}/\text{NH}_4\text{Cl}$ (9:1) saturation solution might be used. The efficiency of the washing could then be checked with AgNO_3 .
- In the present LABEX procedure the entire soil sample is used for the distillation of adsorbed ammonium. However, it is also possible to extract the adsorbed ammonium with a salt solution (e.g. KCl or $\text{Ca}(\text{NO}_3)_2$) and use only an aliquot of this solution for the distillation of the extracted ammonium (FAO Soils Bulletin 10). This allows several analyses if necessary.

Particle Size Analysis (8703-11/12-1)

- In the calculation procedure of the pipette method the sample weight is defined as the sum of the weights of the individual fractions. This implies that the total of the sand, silt and clay is always 100%. To detect errors in procedures it would be better to use the original sample weight (oven-dry basis) corrected for organic matter, carbonates and gypsum. However, when data on the latter are not available the former proposed calculation has to be used.
- In the procedure of the hydrometer method 50 g sample on an oven-dry basis should be used. Consequently the moisture correction factor can be omitted from the calculation whereas "s" (sample weight) has to be replaced by 50.

Phosphorus-Dabin (8703-53-1)

- Erroneously boric acid was omitted from the analytical procedure. As a result sample extracts did not show the blue colour development. Boric acid should be added to the sample extracts after purification and before the addition of the mixed reagent. Boric acid only affects the colour development when fluoride (extracting solution) is present. Standard series without extracting solution and boric acid do show a colour development.
- The standard phosphate solutions and the standard series are all made to volume with distilled water whereas it would be better to use the extracting solution. In addition sulphuric acid, boric acid and mixed reagent should be added to the standard series in order to obtain the same solution matrix as the sample extracts.
- The reaction between phosphorous and molybdate is enhanced by higher temperature (heating at 80°C), and the ratio of the molybdate to the sul-

phuric acid concentration in the mixed reagent seems to be rather critical. Results from participants suggest that this ratio may be too small (low pH) to observe a colour development in the sample extracts.

- Considering all the comments, the analytical procedure will be entirely revised and tested for the use in the next exchange round.

Phosphorus-Bray/Olsen (8703-51/52-1)

- Similar to the modified Olsen procedure (Dabin), the standard solutions and standard series have a different solution matrix as compared with the ample extracts. A comparison among the various existing procedures for each method shows a large variation in the composition of the standard solutions and standard series. Unfortunately it is unknown whether these differences significantly affect the analytical results. For this reason it may be better to use the same solution matrix in the standard series and the sample extracts.

Phosphorus-Blakemore (8703-57-1)

- Often phosphate retention is increased by air-drying soil samples. This is particularly the case with Ando Soils. The procedure for P-retention prescribes the use of air-dry samples. This is in contradiction with the rule that Ando soil samples, in principle, be treated in the field-moist state since so many properties are irreversibly changed by drying. This aspect needs further discussion to arrive at standardization.

Gypsum (8703-42-1)

- The Ca standards have varying amounts of La and these amounts are not the same as the amount of La in the samples. This may affect the measurement of Ca by AAS.
- In the calculation a factor 2, corresponding to the final dilution, should be included. However, if the standard serie undergoes the same final dilution with La as the samples, the factor 2 may be omitted, provided the values 0 to 25 ppm Ca are used.

Final Remarks

- From the preceding comments it is evident that some analytical procedures need to be revised. These revised procedures will be sent to the participants of the next exchange round. A list of revised procedures will be published in the next Newsletter.
- Frequently participants make suggestions for improvements in the LABEX procedures. This is highly appreciated. At the same time we would like to repeat that the choice of LABEX procedures remains arbitrary as they are not meant to become officially standard. Naturally we encourage participants to submit suggestions for improvement and standardization of methods.

2.4 The LABEX soil analytical data

A review of the analytical results from the sample exchange round of 1987 reveals a large number of different data sets (combinations of soil sample and type of analysis). This is caused by the wide choice of available soil samples and analyses. As a result, relatively few data are available for a statistical analysis of the analytical results from each data set. This is

certainly not the aim of the LABEX programme.

In order to obtain a reasonable number of analytical results for each data set, the choice of soil samples and analyses needs to be reduced. For this reason there will be a limited choice of soil samples and analyses in the next exchange round (ref. section 5).

3 Publications from Participants

Recently two publications were received from two participants of the LABEX programme. LABEX gratefully acknowledges the receipt of these publications and all LABEX participants are encouraged to send their (internal) publications or reports on quality control to the LABEX programme secretariat. In this way LABEX may serve as a medium to exchange information on quality control among the participants of the LABEX programme.

"Problems related to texture analysis by the Bouyoucos method" (in Spanish) by Klaudia Oleschko and Jorge D. Etchevers B.
Centro de Edafologia, Colegio de Postgraduados, 56230 Chapingo, Mexico.

Summary

Soil texture analysis is paramount in soil science studies and is conducted on a routine basis in many laboratories. However, procedures commonly used not always yield congruent results. Differences are attributed to variation in the soil dispersion achieved and the method used to measure particle size. The objective of the present work was to compare the routine soil texture analysis used in Mexico (Bouyoucos) with the method proposed by Kachinsky (pipette) and results generated by a soil sample exchange, where a procedure proposed by ISRIC was applied. Soil samples from Mexico and several other countries, representing major soil orders, were analyzed by Bouyoucos, Kachinsky and ISRIC procedures. In general the routine Bouyoucos method yielded the highest clay percentage, however, textural classes determined with data from the three procedures were similar. Higher clay percentages measured with the Bouyoucos method were probably due to soil particles being broken by strong mechanical dispersion. Bouyoucos and ISRIC procedures showed to be inadequate for conducting soil texture analysis in salt affected soils. The percentage of clay in these soils, determined by any of these methods was higher (up to seven times) than the percentage of clay obtained with Kachinsky's method. The presence of soluble salts and carbonates as fine material in suspension may be responsible for this.

"Variability of Laboratory results" (in English)

by Dr R. Breitbart, FAO Soil Chemist.

Soil Mapping and Advisory Unit, UNDP Office, P.O. Box 54, Gaborone, Botswana.

Summary

For the East African Soil Correlation Conference held in Botswana 1987, 21 soil profiles (124 samples) have been described and sampled. Analyses were carried out at the USDA Soil Survey Laboratory in Lincoln, Nebraska (USA) and at the Botswana Soil Survey Laboratory. The analytical data produced by

the two laboratories are compared to obtain information about the reproducibility of analyses. The analyses include:

- pH (H_2O and $CaCl_2$)
- EC (sat. paste extract)
- org.C (Walkley & Black)
- CEC (Ammonium Acetate, pH 7.0)
- Exch. Bases (ibid.)
- Particle Size Analysis (Hydrometer and Pipette Method)

In addition the within-laboratory variability was assessed with a standard sample included in every batch and with a check routine to compare soil parameters and to detect outliers.

4 Alternative CEC Methods

4.1 Introduction

At the LABEX workshop, August 1986, four new procedures to determine the Cation Exchange Capacity and Exchangeable Bases of soils were presented. These methods are:

- | | |
|--|----------------------|
| 1. Choline-Chloride method (ChoChl) | LABEX code 8703-22-1 |
| 2. Cobalti-Hexammine method (CotHex) | LABEX code 8703-23-1 |
| 3. Lithium-Barium-EDTA method (LiEDTA) | LABEX code 8703-24-1 |
| 4. Silver-Thiourea method (AgTu) | LABEX code 8703-25-1 |

It was recommended that these methods, prior to their use in the LABEX programme, be tested by a small number of laboratories. This preliminary test should be used to decide if the procedures are suitable for future testing in the LABEX programme.

In addition to these new procedures also the Ammonium Acetate method (LABEX code 8703-21-1) was used for comparison. The five methods were tested on six LABEX samples, representing a Vertisol (no.17 and 18), a Ferralsol (no.25 and 26) and a Luvisol (no.27 and 28).

The participating laboratories were:

- CSIRO, Division of Soils, Urrbrae, Australia
- New Zealand Soil Bureau, Lower Hutt, New Zealand
- Dept. of Soil Science and Geology, Wageningen, The Netherlands
- ISRIC, Wageningen, The Netherlands
- National Soil Survey Laboratory, Lincoln, USA

Participating in a special programme like this implies a lot of extra work for the laboratories. Therefore we gratefully acknowledge the contribution of these five laboratories. Such cooperation is indispensable for the testing and evaluation of alternative analytical procedures.

4.2 Principle of the Methods

For those who do not have the LABEX procedures of the four alternative methods to determine the CEC and Exchangeable Bases, a brief outline of the principle of each method is given below.

Choline-Chloride method

Exchangeable Bases are extracted with a Choline Chloride, $(\text{OHC}_2\text{H}_4)(\text{CH}_3)_3\text{NCl}$, reagent. The CEC is determined by first saturating the exchange complex with Ca. The excess Ca is removed with polyvinylalcohol. Next, the adsorbed Ca is exchanged with a Choline Chloride reagent and measured in the extract. A washing step for water soluble salts may be included.

Reference: Tucker B.M., 1985. A proposed new reagent for the measurement of cation exchange properties of carbonate soils. *Aust. J. Soil Res.* 23: 633-642.

Cobalti-Hexammine method

In this single step method the adsorbed cations are replaced by Cobalti Hexammine, $\text{Co}(\text{NH}_3)_6^{3+}$. The exchanged Na and K are determined by flame emission and the exchanged Ca and Mg by atomic absorption spectrometry (AAS). The CEC is determined by measuring (AAS) the decrease of Co in the extract.

Reference: Orsini L. and J.C. Remy, 1976. Utilisation de chlorure de cobaltihexammine pour la détermination simultanée de la capacité d'échange et des bases échangeables des sols. *Sciences du Sol. Bull. de l'AFES*, 4: 269-270.
Oliver R., 1984. Étude comparative de deux méthodes d'extraction et de dosage des bases et de la capacité d'échange sur les sols du Sénégal. *L'Agronomie Tropicale*, 39-1: 14-21.

Lithium-Barium-EDTA method

In this single step method the adsorbed cations are replaced by Li and the exchanged Ca and Mg are chelated by EDTA. Determination of the exchangeable cations is done by flame emission (Na and K) and AAS (Ca and Mg). The decrease of lithium in the extract, determined by flame emission, is a measure for the CEC. The procedure includes three modifications: for non-calcareous and non-gypsiferous soils, for gypsiferous soils and for calcareous and gypsiferous soils.

Reference: Begheyn L.Th., 1987. A rapid method to determine cation exchange capacity and exchangeable bases in calcareous, gypsiferous, saline and sodic soils. *Commun. in Soil Sci. Plant Anal.*, 18(9): 911-932.

Silver-Thiourea method

In a single step centrifuge extraction the adsorbed cations are replaced by silver thiourea, $\text{Ag}(\text{SCN}_2\text{H}_4)_n^+$. The exchanged Ca and K are determined by flame emission and the exchanged Ca and Mg by AAS. The CEC is determined by measuring the decrease of Ag in the extract by AAS.

Reference: Chabbra R., J.L. Pleijsier and A. Cremers, 1975. The measurement of the CEC and Exchangeable Cations in soils: a new method. *Proc. Int. Clay Conf.*, Mexico, 1975: 439-449.
Pleijisier, J.L. and A.S.R. Juo, 1980. A single-extraction method using silver-thiourea for measuring exchangeable cations and effective CEC in soils with variable charges. *Soil Sci.* 129:205-211.

4.3 Preliminary Results

All the analyses were performed in duplicate by four of the five participating laboratories. Unfortunately not every analytical method was tested by all the five participants. Two methods (CholChl and LiEDTA) were performed by only three laboratories. One method (AgTu) was tested by four laboratories and the other two methods (Amm.Ac and CotHex) were performed by all the participating laboratories.

Prior to a simple statistical analysis, the submitted data were checked on errors and outliers. The values from duplicates which differed by more than ten percent were omitted. Outliers designated with the Q-test (Dixon, Methods of Soil Analysis, ASA Monograph 9, Part 1, 1986) were also removed from the original data set.

The remainder was used to calculate the mean, the standard deviation and the coefficient of variation (Cv) for each method and every sample. Finally the 95% confidence limits were calculated to illustrate the effect of the limited number of analyses performed with each analytical method.

The preliminary results from a simple statistical analysis of the analytical data are shown below.

CATION EXCHANGE CAPACITY (me/100 gr)

sample		Amm.Ac	AgTu	ChoChl	LiEDTA	CotHex
Labex 17	MEAN	86.5	87.4	55.9	74.9	89.7
	ST.DEV	6.0	4.4	8.3	8.1	6.1
	Cv (%)	6.9	5.0	14.8	10.8	6.8
	95% C.L.	79.0-94.0	80.4-94.4	35.3-76.5	54.8-95.0	80.0-99.4
Labex 18	MEAN	85.8	93.6	57.0	71.9	94.9
	ST.DEV	5.5	5.0	7.1	7.2	5.8
	Cv (%)	6.4	5.3	12.4	10.0	6.1
	95% C.L.	79.0-92.6	85.7-101.6	39.4-74.6	54.0-89.8	85.7-104.1
Labex 25	MEAN	3.9	4.0	1.8	3.1	2.6
	ST.DEV	0.9	1.0	0.6	0.1	0.3
	Cv (%)	24.3	25.9	25.9	1.6	10.2
	95% C.L.	2.8-5.0	2.4-5.6	0.3-3.3	2.7-3.5	2.1-3.1
Labex 26	MEAN	1.2	1.9	0.9	2.1	0.7
	ST.DEV	0.1	0.6	0.2	0.7	0.1
	Cv (%)	11.8	32.6	28.8	32.6	21.9
	95% C.L.	1.0-1.4	0.9-2.9	0.4-1.4	0.4-3.8	0.5-0.9
Labex 27	MEAN	12.1	14.3	9.9	9.8	11.5
	ST.DEV	0.7	1.4	1.7	1.6	0.9
	Cv (%)	5.6	9.6	16.7	15.9	7.9
	95% C.L.	11.2-13.0	12.1-16.5	5.7-14.1	5.8-13.8	9.3-13.7

Labex 28	MEAN	9.3	11.1	8.5	11.7	10.4
	ST.DEV	0.8	0.7	1.4	1.6	1.1
	Cv (%)	8.9	6.5	16.4	13.3	10.4
95% C.L.		8.3-10.3	10.0-12.2	5.0-12.0	7.7-15.7	8.7-12.1

EXCHANGEABLE Ca (me/100 gr)

sample		Amm.Ac.	AgTu	ChoChl	LiEDTA	CotHex
Labex 17	MEAN	57.54	56.65	40.13	57.63	55.16
	ST.DEV	3.36	6.00	0.13	7.40	6.38
	Cv (%)	5.8	10.6	0.3	12.8	11.6
95% C.L.		53.36	47.11	38.96	39.25	47.23
		61.72	66.19	41.30	76.00	63.09
Labex 18	MEAN	49.89	51.09	30.12	52.57	47.43
	ST.DEV	4.55	7.00	4.96	8.75	5.48
	Cv (%)	9.1	13.7	16.5	16.7	11.6
95% C.L.		44.24	39.97	17.81	30.83	40.61
		55.54	62.22	42.43	74.30	54.24
Labex 25	MEAN	2.24	1.91	1.32	2.42	2.03
	ST.DEV	0.16	0.14	0.55	0.10	0.09
	Cv (%)	7.3	7.2	41.5	3.9	4.3
95% C.L.		1.98	1.69	-0.04	2.19	1.92
		2.50	2.13	2.68	2.66	2.14
Labex 26	MEAN	0.06	0.09	0.04	0.09	0.17
	ST.DEV	0.08	0.01	0.04	0.03	0.03
	Cv (%)	133.3	13.6	100.0	33.3	17.3
95% C.L.		-0.04	0.07	-0.28	-0.18	0.10
		0.16	0.11	0.35	0.36	0.24
Labex 27	MEAN	10.12	10.41	8.80	10.55	9.98
	ST.DEV	1.10	0.25	1.86	0.08	0.38
	Cv (%)	10.8	2.4	21.1	0.7	3.8
95% C.L.		8.75	10.01	4.18	10.37	9.52
		11.49	10.81	13.42	10.74	10.45
Labex 28	MEAN	8.19	8.61	7.40	8.54	8.37
	ST.DEV	0.99	0.33	1.59	0.24	0.27
	Cv (%)	12.1	3.8	21.5	2.8	3.2
95% C.L.		6.96	8.09	3.46	7.94	8.03
		9.42	9.14	11.34	9.14	8.70

EXCHANGEABLE Mg (me/100 gr)

sample		Amm.Ac.	AgTu	CholChl	LiEDTA	CotHex
Labex 17	MEAN	30.56	28.22	21.65	33.68	29.70
	ST.DEV	0.48	0.87	1.40	0.08	5.64
	Cv (%)	1.6	3.1	6.5	0.2	19.0
	95% C.L.	29.81	26.83	18.17	33.00	22.69
		31.32	29.60	25.13	34.35	36.72
Labex 18	MEAN	33.21	31.36	23.42	33.65	31.68
	ST.DEV	1.11	1.20	1.73	9.12	5.76
	Cv (%)	3.4	3.8	7.4	27.1	18.2
	95% C.L.	31.44	29.44	19.12	11.01	24.52
		34.98	33.27	27.73	56.28	38.83
Labex 25	MEAN	0.43	0.37	0.30	0.46	0.43
	ST.DEV	0.03	0.02	0.11	0.04	0.04
	Cv (%)	5.9	6.0	36.6	9.7	9.0
	95% C.L.	0.39	0.33	0.03	0.35	0.38
		0.47	0.40	0.58	0.57	0.48
Labex 26	MEAN	0.02	0.03	0.00	0.05	0.03
	ST.DEV	0.02	0.00	0.00	0.02	0.02
	Cv (%)	122.5	17.7		40.0	72.1
	95% C.L.	-0.02	0.01		-0.13	0.00
		0.06	0.04		0.23	0.05
Labex 27	MEAN	0.87	0.86	0.92	1.30	0.89
	ST.DEV	0.05	0.06	0.02	0.05	0.08
	Cv (%)	5.7	7.3	1.6	3.9	9.3
	95% C.L.	0.79	0.76	0.78	0.85	0.79
		0.95	0.96	1.05	1.75	1.00
Labex 28	MEAN	0.96	0.92	0.75	1.08	0.98
	ST.DEV	0.05	0.05	0.01	0.03	0.08
	Cv (%)	5.3	5.6	0.7	2.3	8.3
	95% C.L.	0.88	0.84	0.70	0.85	0.88
		1.03	1.00	0.79	1.30	1.08

EXCHANGEABLE K (me/100 gr)

sample		Amm.Ac.	AgTu	CholChl	LiEDTA	CotHex
Labex 17	MEAN	1.30	1.37	0.08		1.06
	ST.DEV	0.07	0.19	0.03		0.07
	Cv (%)	5.1	13.8	33.3		6.6
	95% C.L.	1.22	0.90	-0.15		0.95
		1.38	1.83	0.30		1.17
Labex 18	MEAN	1.22	1.21	0.11		0.94
	ST.DEV	0.03	0.20	0.02		0.05
	Cv (%)	2.3	16.3	16.6		5.2
	95% C.L.	1.18	0.72	0.07		0.86
		1.27	1.70	0.16		1.02
Labex 25	MEAN	0.08	0.07	0.06	0.06	0.07
	ST.DEV	0.03	0.00	0.05	0.03	0.00
	Cv (%)	34.2	7.1	83.9	50.0	6.4
	95% C.L.	0.03	0.05	-0.07	-0.21	0.06
		0.12	0.08	0.20	0.33	0.07
Labex 26	MEAN	0.02	0.02	0.02	0.03	0.02
	ST.DEV	0.02	0.01	0.02	0.03	0.01
	Cv (%)	102.0	62.3	100.0	100.0	63.7
	95% C.L.	-0.03	0.00	-0.12	-0.24	0.00
		0.06	0.03	0.15	0.30	0.03
Labex 27	MEAN	0.29	0.18	0.02	0.27	0.19
	ST.DEV	0.02	0.15	0.02	0.04	0.02
	Cv (%)	6.0	83.1	100.0	13.2	10.0
	95% C.L.	0.26	-0.06	-0.12	-0.05	0.16
		0.32	0.41	0.15	0.58	0.22
Labex 28	MEAN	0.21	0.15	0.01	0.07	0.12
	ST.DEV	0.03	0.13	0.01	0.03	0.05
	Cv (%)	13.8	86.9	100.0	42.9	39.6
	95% C.L.	0.18	-0.06	-0.08	-0.20	0.06
		0.25	0.35	0.10	0.34	0.18

EXCHANGEABLE Na (me/100 gr)

sample		Amm.Ac.	AgTu	CholChl	LiEDTA	CotHex
Labex 17	MEAN	0.89	0.56	0.44	0.65	0.71
	ST.DEV	0.11	0.11	0.06	0.05	0.06
	Cv (%)	12.3	19.7	12.3	7.7	8.2
	95% C.L.	0.75	0.29	0.29	0.20	0.64
		1.02	0.83	0.59	1.10	0.78
Labex 18	MEAN	9.18	10.68	7.08	8.76	9.10
	ST.DEV	0.74	0.18	1.22	1.24	1.09
	Cv (%)	8.1	1.6	17.2	14.1	12.0
	95% C.L.	8.26	9.10	4.05	5.68	7.75
		10.11	12.25	10.11	11.83	10.46
Labex 25	MEAN	0.10	0.06	0.13	0.03	0.04
	ST.DEV	0.03	0.01	0.10	0.03	0.02
	Cv (%)	28.3	23.6	78.0	100.0	54.9
	95% C.L.	0.06	0.02	-0.12	-0.20	0.01
		0.14	0.10	0.37	0.25	0.07
Labex 26	MEAN	0.05	0.01	0.08	0.00	0.01
	ST.DEV	0.04	0.01	0.08	0.00	0.01
	Cv (%)	85.8	93.5	100.0		173.2
	95% C.L.	0.00	-0.02	-0.12	0.00	-0.01
		0.10	0.04	0.28	0.00	0.02
Labex 27	MEAN	0.09	0.08	0.01	0.02	0.08
	ST.DEV	0.05	0.03	0.01	0.02	0.04
	Cv (%)	62.4	36.8	100.0	100.0	47.6
	95% C.L.	0.02	0.01	-0.04	-0.16	0.03
		0.16	0.15	0.05	0.20	0.12
Labex 28	MEAN	0.10	0.09	0.00	0.02	0.06
	ST.DEV	0.07	0.02	0.00	0.02	0.01
	Cv (%)	68.0	22.0		100.0	14.9
	95% C.L.	0.01	0.04		-0.16	0.05
		0.18	0.14		0.20	0.07

4.4 Discussion

Before a discussion of the results some general remarks have to be made with regard to the interpretation of the results from the statistical analysis.

The results indicate that the precision of each method (Cv) shows a large variation among the different LABEX samples. This variation in precision is strongly related to the magnitude of the parameter to be analyzed. Samples with very low values for the CEC and Exchangeable Bases, generally also show the lowest precision. Even a small measurement error will greatly affect the precision of the analysis. This becomes evident if we compare the results from LABEX samples 25 and 26 with those from the other samples. Samples with very low values of the parameters to be analyzed are not suitable for the use as standard samples or to test analytical methods. This should be noted when standard samples are selected.

Due to the limited number of analyses (three), the results obtained by the CholChl- and LiEDTA methods should be interpreted with caution. Different methods may attain the same levels of precision although they are estimated from a different number of analyses. However, the confidence limits will show an increase with decreasing sample size. This is demonstrated by (too) large confidence limits despite an acceptable precision level.

Another source of errors is introduced by the inexperience of participants with new analytical methods. This is probably one of the reasons for the relative good performance of the Amm.Ac. method. For future testing of alternative analytical procedures it may be better to invite also laboratories which are already familiar and experienced with the particular methods.

Cation Exchange Capacity:

Among the different CEC procedures, the Amm.Ac.-, the AgTu- and the CoHex methods attain a relatively high level of precision (5-10%). The corresponding confidence limits show a rather large variation. The other two methods (CholChl and LiEDTA) show a lower level of precision and larger confidence limits.

Except for the CholChl method, the CEC values obtained with the other alternative methods compare fairly well with those obtained with the Amm.Ac. method. The CholChl method gives consistently lower CEC values than the other four methods.

Considering the limited number of analyses, it is not warranted to draw final conclusions with respect to the performance of the different methods. A valuable comparison of the different methods would require much smaller confidence limits (more analyses on each sample).

Exchangeable Bases:

Because of the low values of exchangeable Na and K, only the results from exchangeable Ca and Mg are suited to evaluate the performance of the different analytical methods. The Amm.Ac.- and the AgTu method appear to attain higher levels of precision and smaller confidence limits compared with the other methods. Surprisingly, all methods show a higher level of precision for exchangeable Mg.

Similar to the CEC, the CholChl method gives consistently lower values of exchangeable Ca and Mg than the other methods. The values of the exchangeable Ca and Mg obtained with the other methods agree fairly well with those of the Amm.Ac. method.

General:

Despite the limited number of analyses performed with each alternative method, the values obtained with the different methods appear to agree fairly well. However, more analyses are required for each method in order to decrease the 95% confidence limits and to obtain a better estimate of the mean value and the precision level of the method. Once this is achieved, a more reliable comparison can be made between the performance of the different methods.

The final choice of alternative methods for future testing in the LABEX programme also depends on aspects such as tediousness time, labour, costs and equipment required to perform an analysis on a routine basis. These aspects will be covered in the next LABEX Newsletter.

A final report on the alternative methods for CEC and Exchangeable Bases will be prepared in close cooperation with the five participating laboratories. In this report the alternative methods will be evaluated and recommendations will be given for future testing in the LABEX programme.

5 Soil Sample Exchange 1988/1989

5.1 Provisional Planning

The (tentative) timetable for the next exchange round will be as follows:

1. November 1988 : Mailing of Newsletter no.4 and sample list for exchange round 1988/1989, to be returned before December 1, 1988. Participants are also requested to inform the LABEX secretariat if they have the necessary LABEX documentation (Data sheet and original LABEX procedures).
2. December 1988 : Mailing of Newsletter no.5 and revisions of the LABEX analytical procedures. The original LABEX documentation will only be mailed to participants who have requested it.
/ January 1989 Mailing of the LABEX soil samples for the exchange round 1988/1989 to those participants whose order form we have received before December 1, 1988.
3. July 1, 1989 : Deadline for submitting the analytical data (in duplicate on the standard data sheet) to the LABEX secretariat. Please indicate on the data sheets which procedures were used (LABEX or own procedures).
4. September 1989: Mailing of the Interim Report with the results from the Soil Sample Exchange 1988/1989.

5.2 Aims

The main aims of the LABEX Soil Sample Exchange 1988/1989 are:

1. To enable the participants to check their laboratory performance. Participants are able to compare their analytical results with those from other participants and those obtained with standard methods. The results of all the participants, using the LABEX or their own proce-

dures, are listed in the Interim Report.

2. To assess the between-laboratory variability.

Using the analytical results obtained with the 'standard' LABEX procedures, the variability of the analytical results among different laboratories may be estimated. This requires a large number of data from each analytical method performed on each sample (data set). Unfortunately the size of the data sets from the preceding exchange rounds were rather small. This was mainly caused by a large choice in available soil samples and analyses. For this reason the choice will be limited in the 1988/1989 exchange round.

3. To assess the within-laboratory variability.

Preliminary results from the preceding exchange rounds (Pleijssier, 1988) indicate that despite the use of 'standardized' LABEX procedures, the between-laboratory variability is still very high. This is probably due to a high within-laboratory variability. One of the reasons for this may be the fact that participants are not routinely familiar with the LABEX procedures.

The within-laboratory variability will be estimated with the results from replicated analyses of selected standard samples issued by the LABEX programme. Preferably these standard samples should be included in routine analyses throughout the year. For a statistical analysis a minimum number of seven replicates will be required.

A proper evaluation of both laboratory variabilities would require the use of standard analytical procedures. However this may not be feasible as most participants use different methods for their routine analyses. So although LABEX procedures are preferred, the participants are free to use their own analytical procedures for the analysis of the standard samples.

Methods to improve the performance of individual laboratories are not included in the programme of the Soil Sample Exchange 1988/1989. This is simply because we first have to determine the variability within each laboratory. Once this has been established and a comparison with other laboratories has been made, the laboratory performance can be evaluated. Depending on this evaluation a strategy for quality control may be chosen. This may involve the use of several statistical methods to control the quality of analytical procedures (e.g. ruggedness test and control charts). It is evident that this requires an individual approach focussed on the specific problems encountered in each laboratory. The LABEX programme will assist and advise individual participants on methods to improve their laboratory performance.

5.3 Organization

-Analyses-

The participants are requested to perform their analyses (in duplicate) according to their own analytical procedures and according to the LABEX analytical procedures. Please indicate on the datasheets which analytical procedures were used.

Five different types of analyses are made obligatory for all participants. Apart from these compulsory analyses, a limited number of analyses is made optional. The following analyses are available:

Compulsory Analyses (LABEX code) :

1. pH in H₂O and KCl (01)
2. Particle Size Analysis with the pipette (11) or hydrometer method (12)
3. Organic Carbon (31); Carbonates (41) and Gypsum (42) only if present
4. CEC and Exchangeable Bases with ammonium acetate (21)
5. Exchangeable Acidity and Aluminium, if relevant (26)

Optional Analyses (LABEX Code) :

6. Particle Size Analysis with the hydrometer (12) or pipette method (11)
7. Nitrogen (Kjeldahl procedure, in preparation)
8. P-Bray 1 for acid soils (51) and P-Olsen for other soils (52)

-LABEX Soil Samples-

Similar to the analyses there is a limited choice of LABEX Soil Samples available for analysis. Five soil samples are compulsory whereas three samples are optional. These LABEX Soil Samples are the following:

Compulsory Soil Samples :

Major Soil Group	Horizon	Carbonates	Gypsum	Salt	Code
1. Ferralsol	A	-	-	-	88-1
2. Acrisol	B	-	-	-	88-2
3. Phaeozem	A	-	-	-	88-3
4. Vertisol	B	?	-	-	88-4
5. Fluvisol	C	+	-	-	88-5

Optional Soil Samples :

6. Podzol	B	-	-	-	88-6
7. Solonchak	B	+	-	+	88-7
8. Luvisol	B	-	-	-	88-8

-LABEX Standard Samples-

In addition to the regular LABEX samples, standard samples (not certified reference samples or standards) will be issued among the participants of exchange round 1988/1989. The aim is to assess the within-laboratory variability of participating laboratories using a limited number of their own analytical procedures performed on one or two selected LABEX standard samples. A minimum number of seven replicates is required for each analysis on every standard sample. These standard samples should be included as 'hidden blanks' in routine analyses throughout the year. The analytical results from the selected standard samples should be submitted to the LABEX secretariat before July 1, 1989. Of course these data will remain anonymous. The participants are also kindly requested to enclose with these data, a (short) description or reference of the analytical procedure used.

In order to simulate the daily routine performance of a laboratory, the participants will have a limited choice of analyses according their own

procedures. It may be clear that preferably those analyses should be chosen which are frequently performed. A selection has to be made from the following analyses:

- Particle Size Analysis
- CEC and Exchangeable Bases
- Exchangeable Acidity and Aluminium
- Organic Carbon
- Nitrogen
- Phosphorus

There are no restrictions for the number of different analyses that may be chosen. The only limitation is the amount of standard sample available. So participants have to check the amount of standard sample required for their selected analyses (minimal seven replicates !) with the available amount of the standard sample. From the available eight LABEX soil samples of the Soil Sample Exchange 1988/1989, maximum two standard samples can be chosen.

We suggest to select one sample with a high and another with a low value for the parameter to be analyzed. The use of standard samples with very low values for the selected parameters should be avoided because already a small measurement error would have a too large effect on the precision of the analytical method (ref. section 4.4).

Although it would be better to use a fixed set of standard samples, a free choice of standard samples will enable the participants to use soil samples which they also frequently encounter in their routine analyses.

5.4 Elaboration of the analytical data

The results of a statistical analysis of the data submitted by the participants will be published in an Interim Report. In addition to a presentation of results like in the preceding Interim Reports, a more detailed analysis of the between- and within-laboratory variability will be given.

The analytical data obtained with the LABEX procedures are used to estimate the between-laboratory variability. This will be compared with the 'between-laboratory variability' estimated from the data obtained with own analytical procedures. Finally an attempt will be made to evaluate the precision and accuracy of the LABEX methods used.

The within-laboratory variability is estimated from the replicated analyses of each participating laboratory. For the different types of analyses and soil samples a comparison will be made between the results of the participating laboratories.

PLEASE NOTICE THE TIMETABLE AT PAGE 14

Enclosed with this LABEX Newsletter is the sample list for exchange round 1988/1989. Please return this list before December 1, 1988. The planning of the future activities will proceed according to the tentative timetable listed on page 14 of this LABEX Newsletter.

Labex secretariat:
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6700 AJ Wageningen
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LABEX-01

BULLETIN D'INFORMATION LABEX

No. 2

Septembre 1987

Secrétariat LABEX:
ISRIC B.P. 353
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Pays-Bas.

(this newsletter is also available in English)

Le mot du secrétaire

Depuis la dernière édition du Bulletin d'Information, un certain nombre de choses se sont passées. Tout d'abord, le compte-rendu des derniers ateliers sont imprimés. Un exemplaire n'est qu'un livre, mais 1100 exemplaires représentent 30 paquets stockés sur 2 palettes! Enfin, quelques jours plus tard, tout était trié, mis dans les enveloppes adéquates et porté à la poste. Des exemplaires ont été envoyés à ceux qui ont participé à l'atelier, aux autres participants, et aux instituts et bibliothèques avec lesquels ISRIC échange ses publications.

Des exemplaires supplémentaires sont disponibles pour le modeste prix de 10 dollars US, port compris.

Entre-temps, j'ai reçu un erratum de l'un des auteurs. Si vous en trouvez d'autres, je vous serai gré de me le faire savoir.

J'ai reçu une réaction du Dr. Griepink sur le premier Bulletin d'Information en ce qui concerne le Bureau Communautaire de Référence (BCR) de la Communauté Européenne à Bruxelles. Le BCR a un certain nombre d'échantillons de sols et de vidanges disponibles, certifiés pour leur contenu en Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Se and Zn. Bien que l'analyse de ces éléments soit en dehors du rayon de LABEX, cette information peut vous être d'intérêt.

La section 'Questions et Réponses' n'apparaît pas dans ce numéro par manque de sujets adéquats.

En juin, votre secrétaire a participé à une réunion de l'Organisation Internationale de Standardisation (ISO) du Comité Technique sur la qualité des sols. Ce Comité Technique aborde des sujets aussi divers que la terminologie, la dégradation biologique et l'analyse chimique. Ce dernier sujet est en relation étroite avec LABEX et une telle relation est importante. Vous trouverez un rapport de cette réunion dans ce numéro.

Enfin, ces temps-ci, nous sommes très occupés avec la préparation des échantillons de sols pour le prochain échange. C'est-à-dire moulin, tamiser et mélanger les matériaux séchés, ce qui fait beaucoup de poussière. Après cela, chaque échantillon est emballé dans de petits sacs en plastique qui sont fermés hermétiquement. Comme le nombre de participants augmente, la quantité de travail augmente également. Malgré cela, rassurez-vous, je ne m'en plains pas! Au contraire, un programme Labex qui progresse est une reconnaissance de notre travail, ce qui nous convient parfaitement.

L.K. Pleijsier

Erreur dans le compte-rendu de l'atelier LABEX

Mr. Begheyn m'a signalé deux erreurs dans son article sur l'analyse de la CEC avec le LithiumEDTA (page 46). Premièrement, l'adresse correcte de son institut est la suivante: Department of Soil Science and Geology, Agricultural University, Wageningen, The Netherlands.
La seconde erreur est plus grave: page 47, la seconde équation devrait être:

$$K = (MY) / (M).(Y)$$

C'est l'inverse de ce qui est mentionné dans le compte-rendu. A la page 49, après la première equation, il est écrit "(in extract)". Ceci devrait être "(mole ratio in extract)".
La remarque concernant son adresse se rapporte bien sûr aussi à son article sur l'analyse du Carbone Organique page 124.

Echange d'Echantillons de Sols 1987

Emploi du temps:

Comme vous l'avez sans doute remarqué, l'emploi du temps pour l'échange d'échantillons de sols 1987, comme indiqué dans le dernier bulletin d'information, était très optimiste, et réclame de ce fait des modifications.

Nous pensons pouvoir envoyer ce bulletin d'information en Septembre 1987. Le formulaire de demande des échantillons y sera joint.

Les échantillons vous seront envoyés quand nous aurons reçu votre formulaire de demande, probablement au cours du mois d'octobre. Les résultats devront nous être retournés environ 6 mois après, c'est-à-dire au cours du premier trimestre 1988. La date exacte avant laquelle les résultats devront être retournés, sera annoncée en temps voulu.

Echantillons:

Pour l'Echange 1987 nous avons 10 échantillons disponibles. Quelques-uns sont tout neufs, d'autres ont déjà été employés dans l'un des échanges précédents. Sur la liste de la feuille séparée, les données analytiques sont resumées. Ces données sont seulement des indications approximatives sur les paramètres du sol et veulent vous donner une idée de la nature des sols. Il est bien sûr parfaitement possible que vos résultats diffèrent de ceux de la liste.

La partie inférieure de la liste d'échantillons est un formulaire de commande. Veuillez cocher sur ce formulaire les échantillons que vous souhaitez analyser, et renvoyez-le nous. N'oubliez pas d'indiquer votre nom et/ou institut et pays. Nous n'enverrons les échantillons qu'après avoir reçu votre formulaire. L'adresse à laquelle ils seront envoyés sera la même que celle utilisée pour ce bulletin d'information. Dans le cas où elle devrait être corrigée, veuillez nous retourner cette étiquette avec les corrections nécessaires.

Nous vous serions reconnaissants de bien vouloir nous envoyer les formulaires pour que nous les ayons au plus tard le 31 octobre 1987.

Les échantillons seront stérilisés aux rayons gamma avant d'être envoyés. Un certificat de ce traitement vous sera envoyé pour vous faciliter l'importation des échantillons à votre office de douane ou de quarantaine.

Paramètres des sols devant être analysés:

Les paramètres des sols devant être analysés sont ceux dont les méthodes sont fournies. Vous en trouverez une liste sur la feuille accompagnant les méthodes.

Méthodes analytiques:

Pour améliorer la comparabilité des données, il est nécessaire d'utiliser les méthodes analytiques LABEX. Si vous devez vous écarter de cette règle, par manque d'appareils appropriés, de réipients en verre ou de produits chimiques, cela est possible, à condition que vous le mentionniez sur le formulaire de données. Dans ce cas, vos données seront traitées séparément.

Certains participants pourraient penser que les méthodes qu'ils utilisent habituellement donnent de meilleurs chiffres, et que, pour cette raison, ils préfèrent les utiliser. Cependant, il est nécessaire de souligner que le but de LABEX est de réduire la variabilité dans les données analytiques par la standardisation des méthodes. Cela permettra une comparaison mutuelle des données entre les participants. Seuls les participants qui n'ont pas la possibilité de suivre les méthodes LABEX et qui autrement ne pourraient fournir aucune donnée, peuvent s'écarter des méthodes analytiques de LABEX.

Report des résultats:

Pour le report des données, un duplicata des résultats est nécessaire, comme convenu lors des derniers ateliers. Ces duplicatas doivent être des 'duplicatas authentiques'. C'est-à-dire que l'analyse dans son entier, du sous-échantillonnage au relevé de l'appareil, doit être dupliquée et non uniquement la dernière étape avec le relevé.

Le formulaire sur lequel vous devez inscrire vos chiffres suivra en temps voulu, envoyé avec l'un des prochains Bulletins d'Information.

Méthodes Analytiques Provisoires

Certains participants ont déjà commenté les méthodes envoyées en juin dernier. Ces commentaires seront utilisés pour améliorer les méthodes. Cela permettra que, au cours du prochain échange, les méthodes analytiques soient au moins compréhensibles pour tous les participants. Un certain nombre de méthodes sont jointes à ce Bulletin. Les commentaires sont toujours les bienvenus. Veuillez nous renvoyer vos remarques aussi vite que possible.

En ce qui concerne les méthodes alternatives pour la CEC, il peut être intéressant que vous sachiez que la méthode au choline chloride, la méthode au cobalti hexamine, la méthode au lithium barium EDTA et la méthode au silver thio urea sont actuellement testées par un petit groupe de participants LABEX. Comme ces méthodes sont relativement nouvelles et inhabituelles, il avait été conseillé au cours des discussions de l'atelier, de les tester avant de les utiliser pour LABEX. Nous vous tiendrons bien sûr informés des résultats de ces tests dès qu'ils seront disponibles.

Comité Technique ISO sur la Qualité des Sols
Deuxième réunion à Milan (Italie), juin 1987

Qu'est-ce qu'un Comité Technique ISO et quelle est sa raison d'être?

En 1986, l'Organisation Internationale de Normalisation (ISO) a établi un Comité Technique (TC) sur la qualité des sols. Le but de ce TC est de standardiser la terminologie, les analyses (chimiques, physiques et biologiques) et les méthodes d'échantillonnage, toutes relevant du domaine de la qualité des sols. Cette initiative est issue du besoin éprouvé par des spécialistes de l'environnement pour des statuts et règlements internationaux en ce qui concerne la pollution des sols. Des règlements internationaux exigent une comparabilité internationale des données analytiques, obtenues avec des méthodes standards. L'aspect de la pollution du sol est apparent dans l'attention portée à des sujets tels que métaux lourds, pesticides, hydrocarbures etc. Un comité technique ISO ne définira pas de limites d'acceptabilité pour les contaminants. Seules la terminologie et la méthodologie seront normalisées.

Qui participe aux travaux du comité technique ISO?

Les membres de l'ISO sont des instituts nationaux de normalisation, tels que le DIN pour l'Allemagne Fédérale, le BSI pour le Royaume Uni ou l'ANSI pour les U.S.A. Les membres peuvent exprimer leur désir de participer à un comité technique. Actuellement il existe environ 200 comités techniques. Le comité sur la qualité du sol est le TC 190. A l'intérieur du TC 190, 11 pays participent en tant que membre-P (membre participant) et 18 en tant que membre-O (membre observateur). Les membres-P sont: l'Autriche, la Belgique, la France, l'Allemagne Fédérale, la Hongrie, l'Inde, l'Italie, la Jamaïque, le Kenya, les Pays-Bas, et le Royaume Uni. Les membres-O sont: l'Australie, le Canada, le Chili, la Chine, la Tchécoslovaquie, le Danemark, l'Egypte, la Grèce, la Malaisie, le Mexique, la Roumanie, l'Afrique du Sud, l'Espagne, la Suède, la Suisse, la Turquie, les Etats-Unis et la Yougoslavie. En général, les membres se réfèrent à des experts d'universités nationales ou d'instituts de recherche pour les questions spécifiques des thèmes de travail.

L'Association Internationale de la Science du Sol (AISS) a un contact officiel avec ce TC. Cela implique que l'AISS est tenue au courant des développements.

A la réunion du TC à Milan, en juin 1987, les pays suivants étaient représentés: la France, l'Allemagne Fédérale, la Hongrie, l'Italie, les Pays-Bas, la Suède, le Royaume Uni. Parmi les organisations de liaison, seule l'AISS avait envoyé un représentant, Ir. L.K. Pleijsier (au nom du Dr. W.G. Sombroek, Secrétaire-général de l'AISS).

Thèmes de travail du TC 190

A la seconde réunion du TC 190 en juin 1987 à Milan, les thèmes de travail ont été attribués aux sous-comités. Les secrétaires des sous-comités ont été nommés et les animateurs des groupes de

travail ont été désignés. Ces animateurs auront pour tâche d'être la force motrice des travaux du groupe de travail. Les sous-comités sont les suivants:

TC/ Sous-comité:	secrétaire:	thème de travail:
TC 190-SC 1	AFNOR(France)	terminologie et codification
TC 190-SC 2	DIN(RFA)	échantillonnage
TC 190-SC 3	DIN(RFA)	méthodes chimiques et caractéristiques des sols
TC 190-SC 4	BSI(RU)	méthodes biologiques
TC 190-SC 5	NNI(NL)	méthodes physiques

Les thèmes de travail du sous-comité 3 ont une base commune avec LABEX. Ce sous-comité regroupe les groupes de travail et animateurs suivants:

GT:	animateur:	thème de travail:
SC 3/WG 1	Dr. W. Bitter (RFA)	métaux lourds, As, Se
WG 2	Dr. W. Huber (RFA)	composants azotés
WG 3	?	composants sulphureux
WG 4	Mrs.Dr.A.Horvath (H)	cyanure
WG 5	Dr. A. Barbera (I)	composants phosphoreux
WG 6	Dr. K.G.Liphard (RFA)	pétrole
WG 7	Dr. W. Ebing (RFA)	pesticides chlorures, PCB's, hydrocarbures
WG 8	Dr. V. Houba (NL)	chlorures conductivité électrique, pH, potentiel de redox, CEC
WG 9	?	analyse de la taille des particules

Pour nous, les groupes de travail 5, 8 et 9 sont importants. Au cours des discussions du sous-comité et du groupe de travail, des efforts ont été faits pour identifier les pièges de la standardisation.

Quels sont les avantages de ce TC pour LABEX?

Dans la période à venir, les animateurs des groupes de travail vont commencer la rédaction de propositions pour des normes. Pour cela, des normes nationales ou des documents s'y rapportant sont nécessaires. Les méthodes analytiques de LABEX seront également portées à l'attention des groupes de travail. Quelques années seront sans doute nécessaire avant la publication de normes internationales. Les méthodes LABEX pourront alors nécessiter des modifications pour se conformer autant que possible à ces normes.

Cependant, il apparaît sur la liste des membres participants, qu'ils sont principalement de pays Européens, où la pollution des sols est une question politique. Pour cette raison, il est concevable que les normes ISO ne seront appropriées que pour des laboratoires hautement sophistiqués, et limitées aux sols des

climats modérés, de 'type européen'. Dans ce cas, les méthodes LABEX peuvent différer des normes ISO, pour s'adapter à un plus grand nombre de sols et être applicable aux laboratoires sans possibilité d'équipement onéreux. Quoi qu'il en soit, nous suivrons les développements de ce TC de très près. Quand des nouvelles des groupes de travail concernés nous arriveront, nous vous en informerons. Un rapport plus complet de cette réunion du TC 190, également sur d'autres sous-comités, sera publié dans un prochain bulletin de l'AISS.

BCR Matériaux de Référence Certifiés
pour l'Analyse des Eléments sous forme de Trace

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Commission de la Communauté Européenne
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Le contrôle de la qualité analytique nécessite également, en plus d'un contrôle correct dans les laboratoires, une participation dans les inter-comparaisons et, quand ils sont disponibles, l'utilisation des matériaux de référence certifiés. Vu les différences des résultats analytiques souvent rencontrées en science du sol, le Bureau Communautaire de Référence (BCR) a produit et certifié trois sols: un sol calcaire limoneux (BCR CRM 141), un sol légèrement sableux (BCR CRM 142) et un sol amendé aux eaux de vidange (BCR CRM 143). De plus, le BCR a produit trois matériaux de référence pour les eaux de vidanges.

Tous ces matériaux ont été certifiés pour les contenus totaux des éléments présents sous forme de traces (par exemple, Cd, Hg, Pb, Zn, Ni ...). Le tableau ci-dessous donne une liste des éléments et des contenus certifiés.

La préparation, l'homogénéité et les études de stabilité ainsi que les analyses de certification sont données en détail dans un rapport de certification qui est transmis avec l'échantillon de sol.

Deux publications couvrent ce sujet,
sur les sols:

- B. Griepink, H. Muntau, H. Gonska, E. Colinet:
Fres.Z.Anal.Chem. (1984) 318:588-591

sur les vases:

- B. Griepink, H. Muntau, E. Colinet:
Fres.Z.Anal.Chem. (1984) 318:490-494

Echantillons BCR certifiés

SOLS:

CRM 141 limon calcaire	CRM 142 légèrement sableux	CRM 142 sol amendé aux eaux de vidange
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	microgramme/gramme	microgramme/gramme	microgramme/ gramme
Cd	0.36 \pm 0.10	0.25 \pm 0.09	31.1 \pm 1.2
Co	(9.2)	(7.9)	(11.8)
Cr	(75.0)	(74.9)	(228)
Cu	32.6 \pm 1.4	27.5 \pm 0.6	236.5 \pm 8.2
Hg	0.0568 \pm 0.0043	0.104 \pm 0.012	3.92 \pm 0.23
Mn	(547)	(569)	(999)
Ni	(30.9)	29.2 \pm 2.5	99.5 \pm 5.5
Pb	29.4 \pm 2.6	37.8 \pm 1.9	1333 \pm 39
Se	(0.16)	(0.53)	(0.6)
Zn	81.3 \pm 3.7	92.4 \pm 4.4	1272 \pm 30

soluble en eau régale:

Cd	(0.30)	(0.22)	(31.5)
Cr	(53)	(44.4)	(208)
Cu	(31.2)	(25.3)	(236)
Mn	(512)	(527)	(935)
Ni	(28)	(28.9)	(92.7)
Pb	(26.3)	(30.9)	(1317)
Zn	(70)	(80)	(1301)

Matrix	milligramme/gramme	milligramme/gramme	milligramme/ gramme
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SiO ₂	(425.8)	(682.2)	(427.2)
CaO	(179.8)	(49.4)	(93.5)
MgO	(11.9)	(10.9)	(49.0)
Al ₂ O ₃	(105.6)	(94.8)	(101.3)
TiO ₂	(4.7)	(6.2)	(6.7)
Fe ₂ O ₃	(37.4)	(28.0)	(37.5)
P ₂ O ₅	(1.6)	(2.2)	(9.1)
Na ₂ O	(4.3)	(9.7)	(4.1)
K ₂ O	(15.6)	(24.1)	(16.2)

perte à 900 °C	0.2065	0.0853	0.2434
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VIDANGE:

	CRM 144 eaux de vidange origine domestique	CRM 145 eau de vidange	CRM 146 eau de vidange origine indust.
	microgramme/gramme	microgramme/gramme	microgramme/ gramme
As	(6.7)		
Cd	4.82 \pm 0.97	18.0 \pm 1.2	77.7 \pm 2.6
Co	9.06 \pm 0.60	8.38 \pm 0.71	11.8 \pm 0.7
Cr	(485.4)	(105.4)	(784)
Cu	713 \pm 26	429 \pm 10	934 \pm 24
Hg	1.49 \pm 0.22	8.82 \pm 0.88	9.49 \pm 0.76
Mn	449 \pm 13	241 \pm 12	588 \pm 24
Ni	942 \pm 22	41.4 \pm 2.4	280 \pm 18
Pb	495 \pm 19	394 \pm 15	1270 \pm 28
Se	(2.3)	(3.3)	(1.7)
Zn	3143 \pm 103	2843 \pm 64	4059 \pm 90

soluble en eau régale:

Cd	(3.6)	(16.8)	(76.5)
Cr	(494)	(85.2)	(769)
Co	(8.6)	(6.8)	(9.9)
Cu	(694)	(415.9)	(921)
Mn	(436)	(219.6)	(555)
Ni	(947)	(38.5)	(269)
Pb	(479)	(332)	(1255)
Zn	(3090)	(2772)	(4002)

Matrix	milligramme/gramme	milligramme/gramme	milligramme/ gramme
SiO ₂	(136.4)	(217.0)	(228.0)
CaO	(56.8)	(153.5)	(142.0)
MgO	(9.2)	(31.0)	(33.0)
Al ₂ O ₃	(45.8)	(34.4)	(90.0)
TiO ₂	(1.9)	(3.2)	(29.1)
Fe ₂ O ₃	(63.4)	(13.0)	(26.5)
P ₂ O ₅	(50.8)	(75.3)	(59.0)
Na ₂ O	(4.6)	(2.7)	(3.1)
K ₂ O	(7.8)	(4.9)	(5.8)

perte à 900 °C	0.620	0.451	0.377
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Les valeurs entre parenthèses ne sont pas certifiées.
Le rapport de certification décrit en détail la méthode analytique pour obtenir le contenu soluble en eau régale des éléments.
Les échantillons sont sous forme de poudre dans des flacons contenant approximativement 40 grammes.

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