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Final report 2006

Laboratory support for VNCPC and Hanoi University of Technology (HUT)

Enclosure 1

University of Applied Sciences
Northwestern Switzerland

Laboratory support for VNCPC and Hanoi University of Technology

Enclosure 1

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Content

1 Introduction

Based on the first mission of 2 experts of the Institute of Environmental Technology (FHBB, now FHNW) in Muttenz/ Switzerland - Jürg Walder and Christof Jeiziner/ chemists - from October, 19th to 27th in 2003 a second mission to the laboratory of Institute for Environmental Science & Technology (INEST) and Vietnam National Cleaner Production Centre (VNCPC) of Hanoi University of Technology (HUT)) was planned. At the laboratory of INEST a certification according to ISO 17025 is planned in the nearest future.

The content of the mission report from 2003 was a first evaluation of the status, the needs and improvement options for the arrangement of the daily laboratory work and the laboratory management system including quality control and quality assurance measures.

INEST owns an analytical laboratory with a modern equipment for a wide range of environmental analysis, which is not yet used optimally for different reasons. Partly the instruments were not completely installed and put into operation.

The results of the analyses are in some cases not reliable, there are differences in the results of the determination of one parameter using different methods.

The INEST laboratory obtains requests for analytical determinations over a wide range of environmental analytics, including examinations of soil, water and air. The laboratory staff is also able and equipped for certain on-site measurements in the industry using mobile testing kits and sampling.

Within a relative short time it is planned to establish an own quality assurance system and to realise the certification related to ISO 17025 to ensure and strengthen the position of the INEST laboratory as a competent partner in the field of environmental analysis in Vietnam.

The target of the second FHNW mission is the continuation of the laboratory support for INEST.

This report describes the 1-week course on basic laboratory skills, according to the concept from October, 29th 2004. The course, conducted on-site at INEST in Hanoi, should give further support for the work of INEST laboratory to ensure a successful certification and find options for improvement in laboratory work.

The course was held at INEST from 17 to 21 April 2006, the target group was the operational staff of the INEST laboratory.

1.1 Goals

- Evaluation of the actual status of development of accreditation maturity
- Show the progress in comparison with the mission report from 2003; what is already improved and what is still open to change
- Discussing elementary working techniques with the laboratory staff on-site
- Give references for basic lab working aspects related to good laboratory practice to improve the situation, minimise sources of error in the daily lab work and ensure traceable results, all against the background of planned upcoming certification of the INEST laboratory.
- Strengthen the awareness for influences, which can decrease the quality and reproducibility of analysis results.
- Give support in current preparation of certification of the INEST laboratory.

1.2 Expert

The course was prepared and conducted by

Ms. Antje Langbein / Institute for Ecopreneurship, FHNW (Muttenz)

- Laboratory Technician, Textile engineer and Environmental Engineer
- Assistant at Institute of Environmental Technology and head of institute's laboratory
- Collaborating in different research projects (e.g. in the fields of recovery of heavy metals from hazardous wastes and waste water, support in pilot plant tests, conducting chemical analyses in the lab including reporting and evaluating the results.

1.3 Participants

Persons in charge on the part of INEST / HUT (Hanoi/ Vietnam):

Mr. Huynh Trung Hai (Ph.D)

- Deputy Director and Laboratory Manager of INEST

Miss Doan Thi Thai Yen

- Quality Manager at INEST laboratory

Participants of INEST:

- Several members of INEST laboratory staff

All the members of laboratory staff are concerned with problems related to good laboratory practice and quality control and quality assurance and have to be integrated in all procedures to prepare and reach successfully the ISO 17025-accreditation of the laboratory.

The laboratory staff members are educated as Environmental Engineers or Chemists.

1.4 Course programme

This week-long course (including 5 working days, from 17 to 21 April 2006) was conceived in different modules of topic. The modules are covering all the different aspects related to the basics laboratory concerns, including basic working techniques, laboratory management, elementary aspects of quality control and assurance, health protection and safety at lab working. Further, in the programme not mentioned points, should be discussed on-site, if necessary.

In the programme were considered the elementary concerns of general laboratory work, which are related to every person of the laboratory staff, all .in view of the planned certification to ISO 17025.

The programme was intentionally planned in an open form, to reserve enough space for individual needs, which appears while the visit on-site in the laboratory. Thus, specific selected materials (papers, scripts etc.) will be issued after the course, tailor-made to the needs of INEST and it will be clarified, which kind of information sources are already on-hand at INEST laboratory.

Short overview - course programme

Date	Topic
April, 17th 2006 / monday	▪ Introduction ▪ Materials in the lab
April, 18th 2006 / tuesday	▪ Laboratory equipment
April, 19th 2006 / wednesday	▪ Chemicals in the lab ▪ Wastes and their disposal
April, 20st 2006 / thursday	▪ Basic lab work techniques ▪ Safety at work
April, 21nd 2006 / friday	▪ Quality assurance and quality management in general ▪ Specific quality assurance procedures

2 Modules of the course

In the following, relative to the single topics of modules, all the observations and findings are described and completed with comments and recommendations for improving of the current situation. All the related working stations were visited on-site in the single laboratory rooms and discussed with the course-participants.

2.1 Day 1 - Materials in the laboratory

Plastic containers

For liquid samples are mainly plastic containers, such as used empty bottles, e.g. soft-drink bottles. No regular sample bottles or any containers are bought, for reasons of economy. Thus any empty container, like bottles and bags, made of polymeric materials, are collected. In most cases, if costumer sent a sample for chemical analysis, in the same way samples are filled in such kind of containers.

It has to be take care fort he right pre-treatment of plastic bottles etc. containing liquid samples for metal analysis: rinse well and acidify the liquid sample to pH <2 to avoid precipitations of metal hydroxides and adsorption of metal ions out of the sample onto the wall of the container. It must be considered, that in some cases (storage of samples for determination of some organic parameters, e.g. PCBs, no plastic bottles may be used.

On the other side, there may be not ignored, that the specific properties of polymers (beside the possible adsorption of metal ions or some organic compounds onto the polymeric material) there may be leached out organic parts out of the polymer, e.g. softener or other auxiliaries. This all can influence and falsify the quality of the analytics, particularly in trace analysis.

Laboratory equipment

The most laboratory equipment, like pipettes, beakers, burettes etc., is made of glass. All the glassware is cleaned by hand with conventional detergents and using a brush to remove rough contaminations. No automatic washing machine is available. Pipettes (bulb and graduated pipettes) are cleaned with a mixture of $K_2Cr_2O_7$ and H_2SO_4 (Chromium-sulphuric acid) and rinsed with distilled water. After cleaning, the glassware is dried in one of several drying chambers; mostly parallel to drying of samples for dry-matter determination at 105 °C. In some cases glassware is also dried at higher temperatures, up to 130 °C.

The temperature for drying of glassware may not be chosen higher than 80 °C. The calibrated volume especially in volumetric measuring instruments like pipettes of different kinds may be changed by deformation of the material. Thus it has to be ensured, that glassware only is cleaned at temperatures ≤ 80°C and, additionally, never at the same

time with any samples, to avoid cross-contaminations from them. The best would be to reserve one of the drying chambers only for glassware drying at 80 °C, and to label it separately and clearly.

Chromium-sulphuric acid is toxic for the environment and is rinsed out into the sewage, when it is used as a cleaning agent for pipettes, because of its content of heavy metal. It should be replaced with a heavy metal free alternative. For this purposes can be used a mixture of concentrated H_2SO_4 and H_2O_2 (30 %)- Peroxy-sulphuric acid. For producing it, first give in the Peroxide-solution and then add in carefully the Sulphuric acid. This solution is only for immediate use and has to be prepared always fresh, because of its very short shelf life. The residence time for cleaning glassware is from a few minutes up to 2 hours, always depending on the degree of contamination.

2.2 Day 2 - Laboratory equipment

The properties of the different common laboratory instruments for daily use have been discussed and explained. The descriptions and labelling on the glassware should be considered (calibration conditions e.g. like temperature). The most used kinds of pipettes are bulb and graduated pipettes. 2 air-interface pipettes (from french suppliers) are at hand, but used very rarely, because the pipette tips as expendable are very expensive.

Pipetting aids (pipetting balls) are available and should be always used.

If graduated or bulb pipettes are used, they should be rinsed with the sample 1 or 2 times, to avoid any trouble caused by possible contaminations left in the pipettes. The rinsing liquid (sample) has to be discharged an not given back to the sample container.

Cleaned and rinsed glassware (especially volumetric measurement instruments, e.g. pipettes) may be dried only at temperatures not exceeding 80°C. Otherwise the calibrated volume, labelled on the instrument, is not guaranteed. Glassware should not be dried in the same drying cabinet, which is used for the drying of any sample, because this can cause cross-contamination, which can potentially disturb any analytical actions.

For the drying of glassware a separate drying cabinet should be reserved and labelled.

Grounded connections on glassware have to be greased, where air-tightness is required, e.g. at the grinding on desiccators, which are used for determination of dry matter. Otherwise the conditioned samples can uptake additional humidity from the ambient air (high air humidity). The drying agent ($CaCl_2$) is dried from time to time in the drying cabinet.

Alternative to $CaCl_2$ as drying agent could be used Silica gel as granulate material including a moisture indicator. There are different particle sizes available. The indicator allows a simple optical control of the saturation level by changing its colour.

Contaminated pipettes are actually cleaned using Chromium-sulphuric acid and after acidic pre-cleaning rinsed in several steps with fresh and distilled water.

As an alternative can be chosen KOH-solution (for removal of fatty, oily contaminations) or for other persistent contaminants Peroxy-sulphuric acid, as already mentioned in chapter 2., to avoid heavy metal pollutions into the waste water.

2.3 Day 3 - Chemicals in the laboratory

Chemicals and samples

The choice of the supplier is unavoidable orientated on low prices. The most used chemicals are ordered from Chinese suppliers because of their low prices. But the quality is often less. The labelled properties are often not matched, such as real concentration and purity degree. Thus these chemicals are separately used for educational purposes. For ordered analytics from customers and for research projects at INEST higher quality chemicals from certified suppliers (e.g. MERCK) are ordered. But these chemicals are much more expensive than Chinese products (at factor 10).

In the main laboratory at INEST, where the preparation of samples for the following analytics is carried out, parallel the practical education of students takes place. For educational purposes chemicals of lower qualities are taken, in contrast, for official analytics high quality chemicals, which are certified, are used. It has to be guaranteed, that these 2 kinds of chemical qualities never are changed or mixed, and in this way cause cross contaminations. They should be separated of each other and labelled accurately (e.g. „only for analytics").

At the moment there is not recorded any statistic about the chemical consumption. For evaluation and ordering of the needed chemicals one named person is responsible.

All standard solutions are stored at 4°C in special refrigerators. The labelling of the chemicals seems to exist, but in some cases, safety instructions and hazard symbols are missed. The individual shelf life of self made standards and reagents has to be labelled and considered.

For this reason, the date of production should be labelled additionally. Especially for the preparation of reagents for sample preparation should be take care for proper storage conditions, if it is required (for example $KMnO_4$-solution for sample preparation has to be stored in brown glass bottles, which are closed air-tight, its shelf life is limited to a certain time).

For the preparation of standard solution no written generally admitted instructions (Standard operation procedures/ SOP) are available and have to be established in the future.

Several refrigerators fort he storage of chemicals and standards at 4 °C are available. No food was stored beside the chemicals in them.

Useful information for lab working is described in the MERCK-catalogue „Chemical Reagents 2002", which is available at INEST.

It contents information about laboratory safety instructions, waste disposal and much more useful information for everyday lab working.

Useful information in the MERCK catalogue

Topic	Page number
Description Labelling of chemical bottles, containers etc.	X - XI
Quality degrees of chemicals	XII - XIII
Safety in the lab (general rules)	XVI - XVII
Risk and safety phrases	XVIII - XXI
Safe storage	XXII
Packaging	1231 – 1232
Disposal of chemical wastes	1233 - 1235
Laboratory gases and their physical data	1238 - 1239
pH-Indicators	1240
Solvents, properties and drying	1241
Periodic table of elements	1242

Incoming samples for determination of one or several parameters are stored at 4 °C in a separate fridge with glass doors.

All received samples are labelled and the responsible person (chemist) is deciding, which parameters have to be analysed and what method has to be used. The samples stored in the fridge are sorted by date of incoming (by day of the week).

A long-term storage of samples is not possible because of space problems. After determination of all parameters and sending the results to the costumer, the samples were disposed to the waste. The sample containers are reused, if possible.

For the specific pre-treatment (conservation) of the samples no guide is available. To avoid mistakes in sample preparation, there should be developed SOP's about preparation procedures for all kinds of samples.

The kind of sample treatment has to be clearly recorded. In some cases it is possible, that different persons are working on the same sample, but carrying out different analytics.

Waste disposal

At INEST no own laboratory waste management is present. All wastes are collected in the main laboratory in containers.

Until 2005 INEST was connected for some years to a University-wide programme for yearly free of charge waste disposal. All the collected wastes had to be listed and notified to a central, and then picked up. For the future INEST has to take care for an own waste

disposal concept. The first measure should be to generate a waste statistic and to reduce or avoid chemical and other wastes, because in the future has to be paid fort the disposal. On the other side, there should be produced as less chemical waste as possible, because it is not guaranteed, that the waste is disposed in an environmental friendly way by the assigned company. Probably the waste is actually brought to an open landfill, without any separation of the different kinds of wastes.

Acidic solutions are neutralised with alkali and then given directly into the sewage as an internal disposal measure.

After neutralizing of heavy metal containing acidic solutions a certain amount of heavy metals can be left dissolved in the liquid and is discharged into the environment through the sewage. For this reason,

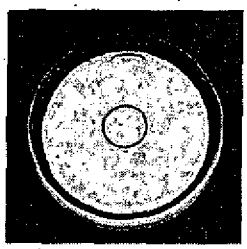
Wastes of solvents are collected in separate bottles. Solid wastes (samples and other chemicals, glass) are collected in containers like board boxes.

2.4 Day 4 - Basic lab work techniques

Different basic lab working techniques are carried out from all laboratory staff members, particularly as preparation steps for the following analytical determination.

2.4.1 Weighing procedure

Analytic balances are placed in different laboratory rooms. For a proper function and trouble-free weighing procedure has to be ensured the exact adjustment of the balance.

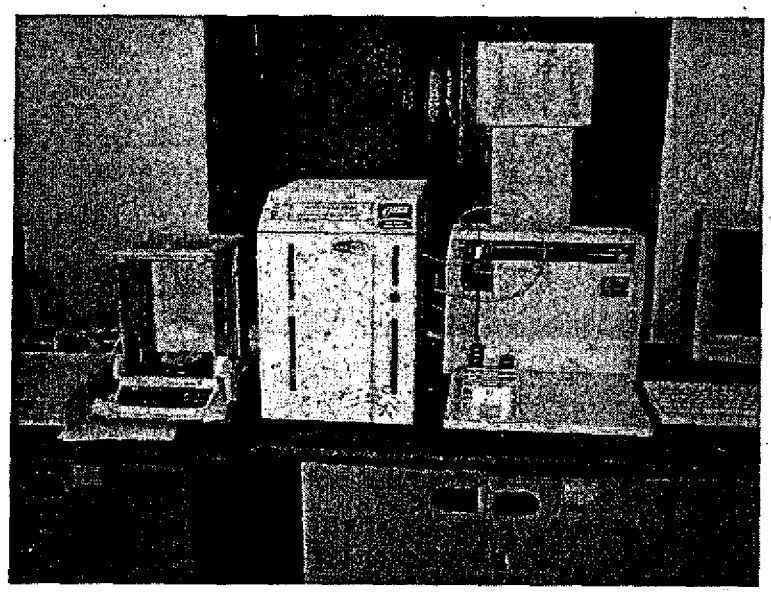


Picture: example for incorrect adjustment

The air bubble is not centered. It has to be placed within the middle ring mark, which is shown in the picture.

The correct adjustment of the balance is conducted by moving the adjusting screws on the stand of the balance.

This should be checked regularly.



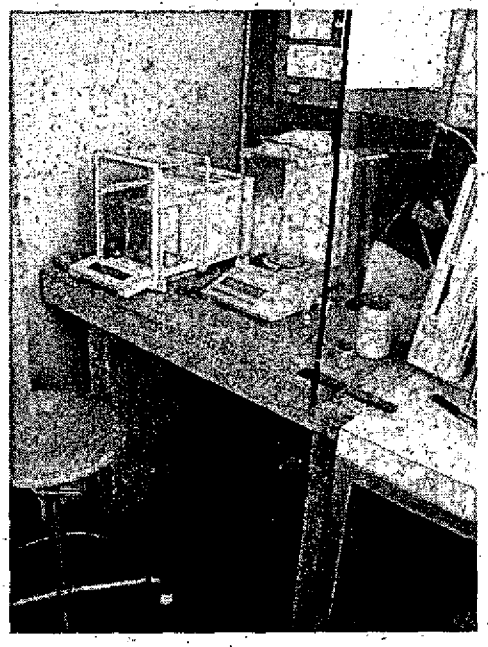
Picture: Analytical balance in the instrument's room at INEST

The balances are standing in laboratory working rooms without any protection from draught, e.g. caused by running air condition.

This can cause problems in balancing procedure (e.g. substance losses) and influence the accuracy of weighted sample, and in following the analytic results.

All balances are positioned on normal working benches and in one case upon a bigger incubation cabinet. Thereby the balances are exposed to the risk of vibrations, what should be avoided.

Ideally balances should be installed in a separate room protected from any draughts and placed on a separate vibration-free fundament (see the following picture).



Picture: working place for balancing at chemical department at FHNW

The balance is placed on a vibration-free fundament.

For additional protection it is surrounded by draught protections (walls and separate shield).

2.4.2 Dry matter determination

This is an often conducted procedure at the INEST laboratory. The balanced samples are packed into usual household aluminium foil and given into the drying cabinet and dried at 105 °C until reaching the constancy of weight after conditioning in the desiccator.

In some cases, washed glassware is dried parallel in the same drying cabinet like used for dry matter determination. These two operations should be separated strictly.

It has to be considered, that the balanced samples never are touched with naked hands to minimise any cross-contaminations.

The desiccators contain $CaCl_2$ as drying agent. This is regenerated by drying in the drying cabinet from time to time. For a better control it is recommended to use Silica gel including a special moisture indicator, which shows by colour change the degree of the saturation level.

The ground surface of the connection between desiccator and its cover has to be greased with special silicone grease to ensure air-tightness of the desiccator.

2.4.3 Volumetric measurement

Standard solutions are stored at 4 °C in fridges. If they are taken out for use, it should be considered to adapt their temperature to the specification of the volumetric measuring instrument (e.g. bulb pipettes).

Self made standards should be labelled with the manufacturing date to ensure the compliance of the shelf life.

2.4.4 pH measurement

In the main laboratory a pH measuring instrument is available and installed on the work bench. As accessory the instrument is completed with a robust polymeric electrode filled with Gel. Calibration standards are available, but there is no official instruction about the intervals, the instrument has to be calibrated.

2.4.5 Combined mobile testing kits (for field tests)

In a separate cabinet are stored several combined testing kits for outside-use and determination of different environmental relevant parameters, e.g. pH, conductivity, dissolved O_2, wind speed, noise, temperature, humidity). The testing kits were not examined.

At INEST laboratory one person is nominated to take care fort he testing kits, to control their completeness and use. Maintenance plans to control the proper function of the kits are not provided.

2.4.6 Distillation of water

For the conditioning of drinking water to cleaned water for analytical purposes 2 distillation devices are available at the main laboratory at INEST:

- One-step distillation unit (water for simple laboratory use)
- Two-step distillation unit for processing water for analytical purposes with higher requirements of purity

Both distillation devices are strongly covered inside with calcification caused by the high raw drinking water hardness. The strong lime layer inside the cooling section (condenser) is inhibiting the thermal exchange between process and cooling water and this reduces the efficiency of the distillation. Simultaneously the quality of the distillate is decreased by potential contaminations out of the limestone residue.

The distillation units should be decalcified continuously using acidic solution (e.g. Acetic acid or Citric acid).

Generally the quality of the distillate can be controlled easily by measuring the conductivity. The equipment for this measurement is available at INEST. Comparable units are delivering a quality, which is described as a conductivity of the distillate ≤ 2 µS/cm.

Other commercial distillation units are including an integrated display for the conductivity. If the conductivity reaches a maximum (for normal analytical purposes 5 µS/cm), an indicator lamp shows that the unit has to be maintained (decalcified and cleaned).

2.4.7 Microwave assisted digestion

For the digestion of samples a laboratory microwave exists. Several digestion beakers made of polymeric material are included as accessory. Probably the instrument's manual is no more complete. The microwave is not often integrated into the laboratory work. Some of the beakers show leakages, and thus it is worried about the suitability for microwave assisted digestion of samples. The partial damages of the beakers and their untightness are caused by overloading the beakers with sample material in earlier times. Samples with a high content of organic compounds (e.g. polymers, sewage sludge) can develop high amount of CO_2 while the digestion process, which can potentially destroy the beakers (e.g. by bursting). Thus the weight of the sample should normally not exceed 500 mg, only for inorganic samples with a very low risk of developing gases while digestion process it is recommended to weight in up to 2'000 mg.

If it is necessary to develop a new digestion method, it is recommended to begin with a minimum of weighted sample, which can be carefully increased.

For the proper function of the instruments all holders of the rotor inside the microwave have to be occupied, empty beakers have to be filled up with blank solution. This is necessary for equable energy and heat distribution.

After the digestion of samples, when the programme is completed, the samples can simply cool down overnight, and be opened without any risk on the next morning, when they are no more under pressure.

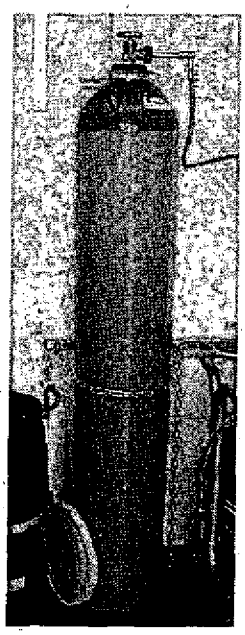
2.4.8 Laboratory gases

Beside the instrument's room in a separated place all the needed laboratory gas bottles are positioned.

The bottles are not locked for a secure stand. A good and simple solution would be to install holders on the walls including chains to secure the bottles and improve the safety.

The gas bottles are transported directly by the supplier to the storage room as shown in the picture.

Gas bottles at INEST laboratory



Holder for gas bottles at FHBB lab

2.4.9 Sterile working bench

On the sterile working bench experiments using Coli bacteria for educational purposes are conducted. The sterile bank includes a UV lamp for disinfection of the working area inside the hood. No additional disinfectants are used.

Generally the space for bacteriological works can be additionally cleaned using alcohol (Methanol or Ethanol, 70% in aqueous solution).

2.4.10 Safety at work

Personal safety measures may not be used in every case, but are available on a minimum level. First aid kits seem to be marginal available, but this was not checked in detail.

At regular intervals all the staff members should be instructed in all the important safety measures (e.g. personal safety and the catalogue of measures in case of fire or any accident) what should be part of implementing a laboratory management system.

2.5 Day 5 - General quality assurance and management

2.5.1 In general

In the nearest future the official certification according to ISO 17025 is aspired. Thus a special responsible person for the own quality management of INEST laboratory is already officially nominated (Quality manager). Its task is to take over the quality system, to take care for the development of the quality system and its implementation, and to instruct the INEST laboratory staff members in the quality system.

At the present time the quality system is in preparation as a pre-stage in view to the planned certification. Some members of the INEST staff were sent to an external training course about ISO 17025 at the end of the year 2005.

The initial certification according to ISO 17025 by an official institution is planned for the end of 2005.

The requests for analytical works (e.g. determination of specified parameters) are incoming and sent to the laboratory manager and then distributed with the samples to the qualified persons, who will do the requested analytics.

The results of the analytics are documented on an official form and signed by the director of INEST and the laboratory manager. Copies are stored in folders in the laboratory manager's office. Often it is not traceable, what happened in detail with the sample. Thus an analysis of error is complicated or impossible in some cases.

Several personal computers are available in the laboratories; one of them is connected to the internet. But they are not connected in a network structure (server), because of the fear of .catching any computer virus and damaging some saved data files.

At the moment the INEST laboratory does not own a laboratory handbook or any established quality system.

The official laboratory handbook of the Institute of Environmental Technology was introduced to the course participants.

All the single chapters and their content were explained and discussed. Examples for form sheets, which are used in the laboratory at IfU and the chemical department of FHBB were shown (form sheet for request of any laboratory work, laboratory journal for description of the current work and the form sheet for analytical results.)

The constitution of the laboratory quality system of IfU (Institute of Environmental Technology, Muttenz, now: IEC/FHNW) is reflected on the electronic folders on the institute's server, where all the laboratory-related information is stored.

Table: content of the IfU laboratory quality system on the server

Topic	Abbreviation*	Content
Correspondence	KOR	Letters, Mails, Fax
Literature	LIT	Electronic files related to laboratory (management, analytics, ...)
Quality Assurance	QS	Lab handbook, form sheets (labels, lab work, ...)
Analytics	ANA	operation instructions (SOP), lab working (order, execution, results)
Administration	VER	Orders, supplier list, waste disposal, instruments, sample management, coordination with chemical department

* In German language

Particularly the scheme for the generation of the registration code-number for laboratory work orders, like described in the laboratory handbook of IfU, was announced.

As an example:

001-05-3510

Serial 3-digit number – 2-digit date (year) – 4-digit internal project number (which the lab work is related to)

Every project at IfU is registered for internal purposes under a 4-digit internal project number.

In a similar way, all electronic documents in the laboratory folder on the server are named according to the following principle:

0000 LAB VER suppliers list_1.7.2005

4-digit internal project number – LAB as abbreviation for the title of the main folder "laboratory" – VER as an abbreviation for the subordinated folders title "Verwaltung" ("maintenance") – short description of the file – additional comments e.g. version number or date of status.

2.5.2 Conducting of analyses

All the analytical instruments at the INEST laboratory are assigned to one person of the laboratory operational staff. The analytical instrumentation is state-of-the-art, in most cases quite new or only in short usage and covers in a wide range all the established methods in environmental analytics.

The tasks of the analytical instrumentation operators are

- operate and calibrate the instrumentation
- record operational parameters
- record results of analytics

The operators are not in every case all-embracing familiar with the instruments and potential of the instrumentation. Probably all the possibilities of the instruments are not yet exhausted. The installation and introduction in usage and handling of the instruments is given by the suppliers and seems to be kept very global. Further consulting service is only available for additional payment.

Normally only the person, who is defined to take care and work with the instrument, is instructed by the supplier. For specific problems the supplier should be contacted for support. Normally the suppliers provide more information about their instruments and development of specific measuring methods. In most cases such information are available from the supplier's internet sites in the form of application reports or other technical documentations. In Vietnam such service is not easily to achieve, because downloads from the internet are limited and not for free.

For the processing of the analysis including preparation of the sample the person working on the related instrument is responsible.

For the determination of parameters mainly reference methods are used, e.g. methods described by EPA (Environment Protection Agency, US), APHA (American Public Health Association, US) and also Vietnamese standards and the usual technical literature about analytical methods is consulted.

In some cases own methods are developed by the operational staff. Specific knowledge about details of analytical methods is not gladly shared with other associates and kept as own knowledge and thus the traceability of analytical results is affected.

To the most measuring instruments are existing books, in which the results of the measurements are enlisted, or in some cases the results are directly filled into computer data files belonging to the instrument's software or in own created Excel-sheets (which allow automatic calculations and additional statistics, if required.

About the specific tests methods, which are used, there are not yet own written down general descriptions or instructions (e.g. in form of a Standard Operating Procedure/ SOP) available.

The documents related to the instruments are not in every case complete, so as seen for example on the BOD-measuring equipment. Application reports with detailed descrip-

tions of analytical methods or about specific analytical problems are not present. So the preparation of own measuring methods can potentially be deficient and complicated.

In some cases are observed varieties of measuring results using different methods for determination of the same parameter. E.g. in the determination of Fe using UV/VIS-Spectrometry and Flame-AAS the results differ. Using Flame-AAS the measured results are higher than compared to Flame-AAS.

Having such differences, several aspects, which all can influence the measuring result, have to be checked for trouble-shooting.

- Proper handling of the instrument (check the instruments parameters and settings; see handbook of the instrument, application reports or comparable literature)
- Measuring method (check of standardisation and calibration)
- Plausibility of the measuring results (check by using the common analytical quality control measures like addition of standard solution to the liquid sample or dilution of the sample in several steps)
- Chemicals and reagents (check quality, shelf life and storage conditions)

Possible reasons for not reproducible measuring results:

- Inappropriate auxiliaries and chemicals in the sample preparation (especially quality, concentration and purity)
- Influences in sample preparation e.g. loss of analyte while digestion of the sample, incomplete chemical reaction in sample preparation, failings in pipetting and volumetric measurement
- Cross-contaminations of the sample or reagents caused by insufficient cleanness.
- Instruments parameter incorrect adjusted (causing lower sensitivity)
- Disturbances in the functionality of the instrument (e.g. plugged sample-inlet tubes causing mismeasurement)

All these points should be clarified to find out the reasons for mismeasurement. An important part beneath the handling of the instrumentation is the understanding of the chemism of the chemical reaction, which is the background of every analytical method, including the preparation of the sample, to facilitate the trouble-shooting.

For every single instrument a handbook with comprehensive descriptions of proper use has to be available and an additional instrument's logbook should be created and established. The logbook should content a maintenance schedule and reporting about the usage of the instrument.

For information about basic procedures of analytical quality control measures the Mission Report from 2003 should be consulted again.

For statistic calculations can be reverted to available analytical literature e.g. „Standard methods for the examination of water and waste water" (APHA), which is available at INES laboratories and contents a separate chapter about statistics) or to the functions of Excel. In most cases, modern analytical instruments include in their software programmes features for statistics or analytical quality control, which can be involved.

Furthermore the continuing education may not be disregarded, as well as the current exchange of experiences with colleagues from HUT or other institution working in environmental analysis.

3 Further Steps ahead to certification

Estimation of actual development status regarding the preparation of accreditation of ISO 17025: Operational procedures are not yet generally admitted described. At the moment, a laboratory handbook is not yet rendered. Several members of the INEST laboratory staff attended a course about ISO 17025 to learn the basics about this norm, and what all has to be included.

Like already mentioned in the Mission Report of 2003, the following steps are necessary:

- Decisions about responsibilities (already done): Quality manager: Miss Doan Thi Thai Yen (directly responsible to the laboratory manager and subordinated responsibilities)
- Development of an own INEST laboratory handbook as a frame: orientated to patterns, definition of aspects to be described
- Constitution of the general format: for traceability reasons and bindingness related information in headline and footer in documents
- Development of SOPs (Standard Operation Procedures), documentation of analytical works and results
- Description of analytical quality assurance measures

All the already existing working procedures at INEST laboratory has to be documented and converted to binding instructions.

In the future the all specific knowledge of every laboratory staff member about the analytical procedures has to be available and traceable by the existence of SOPs.

Although not necessarily required for the accreditation to ISO 17025, general laboratory related aspects should not be disregarded in creating the own laboratory handbook:

- Working safety and health protection
- Waste management (including prevention, collection and disposal)
- Further education of the laboratory staff

The consideration of these additional points helps to improve the working conditions and safety in the laboratory and can help to save costs.

Helpful documents, related to different important aspects of laboratory work and management were collected as electronic files on CD and given over to the INEST laboratory and quality management.