

# **1 Regional Quality Assurance Mechanism**

## **1.1 Objectives**

The TOR for the present assignment simply specify that the Consultant should

"Be responsible for instituting a Regional Quality Assurance Mechanism".

A clarification during the proposal preparation period indicated that LVEMP was referring to the quality of field data.

World-wide experience with all quality assurance systems has shown that quality is not achieved by a top-down control process alone. It requires, most of all, the motivation, engagement and commitment of the staff. Certain tools must be provided to assist and guide the staff, but without the encouragement of the management and the commitment of the staff, the tools are of little use. In the present case, the "tools" are essentially the manuals with instructions for field data collection procedures and laboratory analyses.

## **1.2 Methods**

The methods applied by the Consultant in instituting a QA mechanism were as follows:

- Preparation of a set of field forms with instructions for field data collection and for recording both field data and laboratory analysis results.
- On-board training of field staff in monitoring procedures, methods and routines.
- Checking and adjustment of the laboratory analysis methods.
- Preparation of methods manuals specifically adapted to local conditions in Kisumu and Mwanza.
- Training of laboratory managers in international QA/QC standards and procedures (Kisumu and Mwanza).
- Training of laboratory staff in analyses of water samples for nutrients.

- Comprehensive hands-on training in data validation.
- The first inter-laboratory comparison on accuracy of analyses of nutrient concentrations.

### **1.3 Field Forms**

A comprehensive set of field forms was prepared by the consultant for all the parameters to be measured during the lake monitoring and the monitoring in the catchment. The forms include instructions on measurement procedures and provide a medium for recording all data from field and laboratory. The field forms should "follow the data" from field to the laboratory and to the final user. They are the hard copy of the original, raw data, and should be completed with care and accuracy, and never destroyed.

The field work was divided into a number of programmes as shown in Figure 1.1. During each field trip (corresponding to a programme) measurements were made for a number of different purposes or monitoring tasks, and a field form was prepared for each task.

**Field Programmes - Monitoring Tasks Matrix**

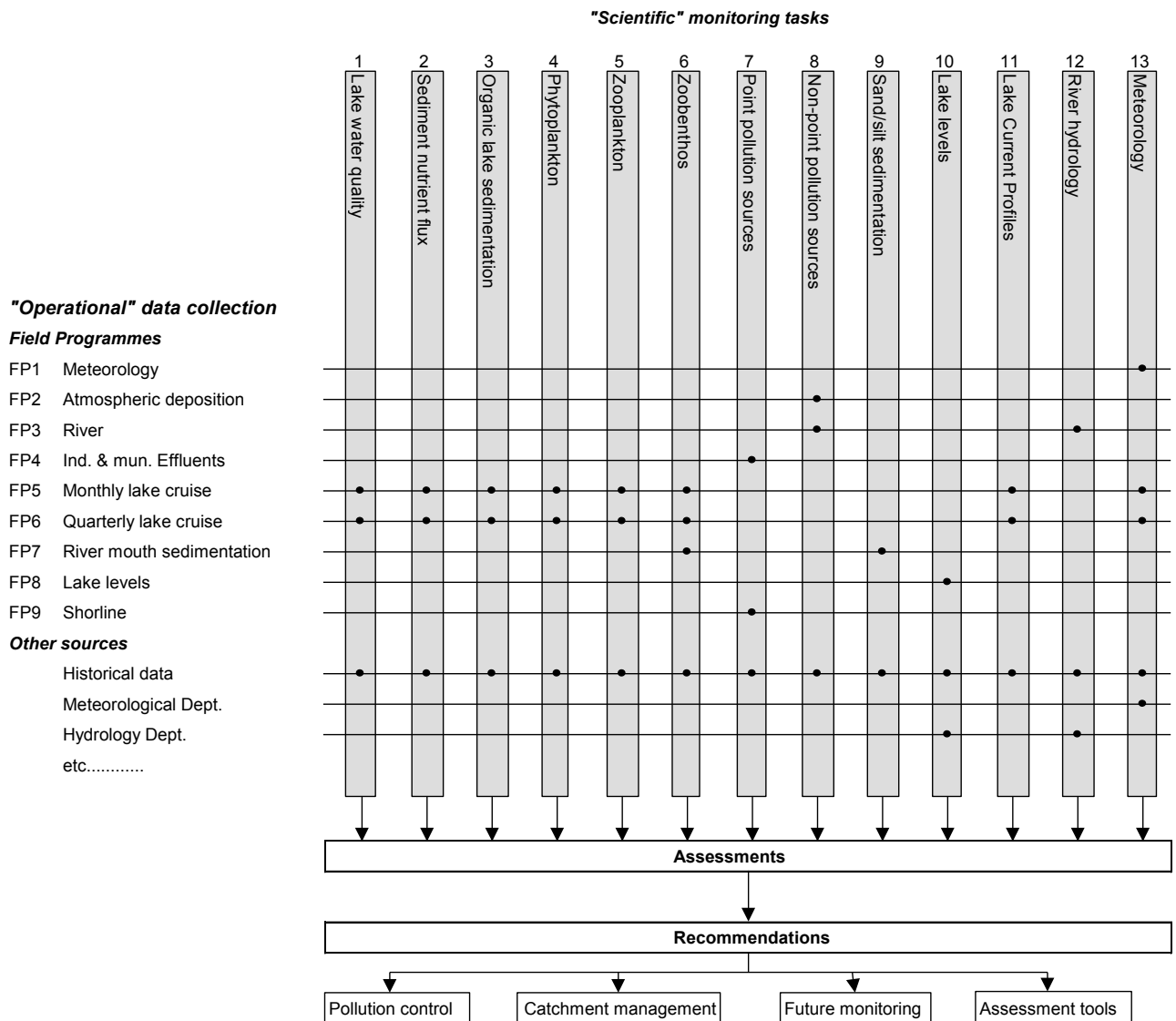


Figure 1.1 Field programmes and monitoring tasks.

A full list of the field forms is shown in T. and some examples of the field forms are shown in Figure 1.2 to Figure 1.5.

Table 1.1 List of Field Forms

| Field Programme                         | Monitoring task  | Data                           | Parameters  |
|---|--|--------------------------------|---|
| Monitoring Tasks                        | Summary of all monitoring tasks and parameters   |                                |   |
| Safety                                  | Safety list for Lake Cruises   |                                |   |
| On-board Monitoring Procedures          | Instructions for monitoring procedures and preservation of samples at each lake station. |                                |   |
| FP1: Meteorology                        | Cover sheet<br>Meteorology   | Field                          | Rain, air temp, humidity, evaporation, solar radiation, wind speed and direction.   |
| FP2: Atmospheric Deposition             | Cover sheet<br>Non-point pollution   | Field<br>Lab                   | Sampling area and volume.<br>TN, TP, NO <sub>2</sub> , NO <sub>3</sub> , NH <sub>4</sub> , PO <sub>4</sub> , Si, PBSi, Alkalinity, TSS, TPC.  |
| FP3: River                              | Cover sheet<br>River hydrology   | Field<br>Lab                   | Gauge height, current profiles.<br>Discharge (m <sup>3</sup> /s), sediment discharge (kg/s), TSS.   |
| FP4: Industrial and Municipal Effluents | Cover sheet<br>Point source  | Field<br>Lab                   | Discharge (m <sup>3</sup> /s).<br>TN, TP, NO <sub>2</sub> , NO <sub>3</sub> , NH <sub>4</sub> , PO <sub>4</sub> , Si, PBSi, Alkalinity, TSS, TPC, sediment content.   |
| FP5: Monthly Lake Cruise                | Cover sheet<br>Equipment list<br>Meteorology<br>Current profile<br>Water quality         | Field<br>Field<br>Field<br>Lab | Rain, air temp, humidity, evaporation, solar radiation, wind speed and direction.<br>ADCP data and unit discharge.<br>Depth, temp, DO, Conductivity, Light, pH.<br>TPN, DON, TN, TPP, DOP, TP, NO <sub>2</sub> , NO <sub>3</sub> , NH <sub>4</sub> , PO <sub>4</sub> , Si, PBSi, Alkalinity, TSS, TPC, Chl-a. |

|                                |   |                                  |   |
|--------------------------------|---|----------------------------------|---|
|                                | Phytoplankton   | Field<br>Lab                     | Depth of sample<br>Species names and counts.  |
|                                | Zooplankton   | Field<br>Lab                     | Depth of sample<br>Species names and counts.  |
|                                | Zoobenthos  | Field<br>Lab                     | Dredge area and sieve data.<br>Species names and counts.  |
|                                | Organic lake sedimentation                                      | Field<br>Lab                     | Trap depths and deployment time.<br>TPP, TPN, PBSi, TPC, DW.  |
|                                | Sediment nutrient flux  | Lab 1<br>Lab 2<br>Lab 3<br>Lab 4 | Sediment oxygen consumption.<br>Nutrient flux: PO4, NO2+NO3, NH4, Si.<br>Pore water nutrient content: PO4, NO2+NO3, NH4, Si.<br>Sediment nutrient content: PO4, NO2+NO3, NH4, Si. |
| FP6: Quarterly lake Cruise     | See FP5.  |                                  |   |
| FP7: River Mouth Sedimentation | Cover sheet<br>Sand/silt sedimentation<br>Zoobenthos - see FP5. | Lab                              | Grain size distribution.  |
| FP8: Lake Levels               | Cover sheet<br>Lake level                                       | Field<br>Lab                     | Raw data<br>Time series.  |
| FP9: Shoreline                 | Cover sheet<br>Point source pollution - see FP4.                |                                  |   |

**Field Programme Sheet****FP5: Monthly Lake Cruise**

| <b>Programming and Staffing</b> |                           | <b>Equipment</b>    |
|---------------------------------|---------------------------|---------------------|
| Frequency:                      | Monthly                   | See list.           |
| Relation to other FP:           | Extended quarterly by FP6 |                     |
| Programming: Week nos.:         |                           | <b>Safety equip</b> |
| Duration:                       | 6-7 days                  | See list.           |
| Scientific/Technical staff:     |                           |                     |
| Support staff:                  |                           |                     |

| <b>Collection of data for monitoring tasks</b> | <b>Sample sets</b>  |
|--|---|
| Task 1: Lake Water Quality                     | S1 WQ profiles (Hydrolab + laboratory)<br>S2 Chlorophyll-a                                    |
| Task 2: Sediment Nutrient Flux                 | S3 Oxygen consumption<br>S4 Nutrient flux<br>S5 Pore water nutrients<br>S6 Sediment nutrients |
| Task 3: Organic Lake Sedimentation             | S7 Sediment traps   |
| Task 4: Phytoplankton                          | S8 Phytoplankton  |
| Task 5: Zooplankton                            | S9 Zooplankton  |
| Task 6: Zoobenthos                             | S10 Zoobenthos  |
| Task 8: Currents                               | S15 Current profiles (ADCP)   |
| Task 13: Meteorology                           | S17 Wind, etc.  |

**Procedures:** See description of Monitoring Tasks and On-board Monitoring Procedures

| <b>Stations</b> | <b>E</b> | <b>N/S</b> | <b>Sample sets</b>                                |
|-----------------|----------|------------|---|
| TP1             |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TP2             |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TP4             |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TP9             |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TP12            |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TP18            |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TL232           |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |
| TL234           |          |            | S1, S2, S3, S4, S5, S6, S7, S8, S9, S10, S15, S17 |

**Remarks:**

Figure 1.2 Field form FP5: Monthly Lake Cruise, Cover sheet.

**FP5: Monthly Lake Cruise****Equipment check List**

| <b>General</b>   | <b>No.</b> | <b>Check</b> |
|--|------------|--------------|
| Field Handbook (forms, program description and sampling procedures).     |            |              |
| Freezer or coolboxes.  |            |              |
| Rope, pencils, markers etc.  |            |              |
| Tool Box   |            |              |
| Measuring tape.  |            |              |
| <b>Current profiling measurement</b>                                     |            |              |
| ADCP or other type of current meter.                                     |            |              |
| ADCP mounting rack.  |            |              |
| External flux gate compass for ADCP.                                     |            |              |
| Laptop PC for ADCP.  |            |              |
| <b>Water quality profile measurements</b>                                |            |              |
| Hydrolab sonde or alternative sondes (Oxygen, light, temp. ph etc.).     |            |              |
| Laptop PC, diskettes.  |            |              |
| Winch with 100 m. wire rope.   |            |              |
| Backup system for Hydrolab.  |            |              |
| Secchi disk + 20m. line with marks for every meter.                      |            |              |
| Glassbottom box.   |            |              |
| <b>Water and plankton sampling</b>                                       |            |              |
| Water samplers (5-8 litres) & 100 m line with meter indications.         |            |              |
| Rinsing bottles (3).   |            |              |
| Measuring tubes (1 l, 100 ml, 10 ml).                                    |            |              |
| Funnels, bucket, plastic cans (3*10 l) & tub.                            |            |              |
| Vacuum pump and flask.   |            |              |
| Aluminum foil and box (for storage of filters).                          |            |              |
| Pre-weighted GFC-Filters for TSS/LOI (1 pr. depth pr. station + extras). |            |              |
| GFC-filters for Chl.a.   |            |              |
| Plastic bottles (3x0.5-1 litres pr. depth and station).                  |            |              |
| 50 µm sieve for zoo-plankton.  |            |              |
| 100 ml. Bottles for zoo-plankton (1 pr. depth pr. station).              |            |              |
| 100 ml. Bottles for Phyto-plankton sample (3 pr. station).               |            |              |
| Distilled water.   |            |              |
| 4% formalin solution.  |            |              |
| Lugols solution.   |            |              |
| <b>Sediment Sampling</b>   |            |              |
| Ekman bottom sampler + 100m. line with marks for every meter.            |            |              |
| 4 tubes (5,2*50cm) & 8 rubber stoppers pr. station.                      |            |              |
| 500 µm sieve for zoo-benthos.  |            |              |
| Beer case for sediment cores.  |            |              |
| <b>Sediment traps</b>  |            |              |
| Line/rope, buoyancy, bottom weight and surface float.                    |            |              |
| 6 traps (bottles, tubes etc.) pr. station.                               |            |              |

Figure 1.3 Field Form FP5: Monthly Lake Cruise, Equipment check list.





|                           |          |                                |                                |
|---------------------------|----------|--------------------------------|--------------------------------|
| <b>Station:</b>           | Date:    | <input type="text" value="/"/> | <input type="text" value="/"/> |
| <b>Position:</b>          | Time:    | <input type="text"/>           | <input type="text"/>           |
| Actual pos: E_____ N_____ | Vessel:  | <input type="text"/>           | <input type="text"/>           |
| WPT No: _____             | Initials | <input type="text"/>           | <input type="text"/>           |

**Laboratory measurements, results:**

| Depth      |  |  |  |  |  | Units | Initials | Approved |
|------------|--|--|--|--|--|-------|----------|----------|
| Sample no. |  |  |  |  |  |       |          |          |
| Lab no.    |  |  |  |  |  |       |          |          |
| TPN        |  |  |  |  |  |       |          |          |
| DON        |  |  |  |  |  |       |          |          |
| TN         |  |  |  |  |  |       |          |          |
| TPP        |  |  |  |  |  |       |          |          |
| DOP        |  |  |  |  |  |       |          |          |
| TP         |  |  |  |  |  |       |          |          |
| NO2        |  |  |  |  |  |       |          |          |
| NO3        |  |  |  |  |  |       |          |          |
| NH4        |  |  |  |  |  |       |          |          |
| PO4        |  |  |  |  |  |       |          |          |
| Si         |  |  |  |  |  |       |          |          |
| PBSi       |  |  |  |  |  |       |          |          |
| Alkalinity |  |  |  |  |  |       |          |          |
| TSS        |  |  |  |  |  |       |          |          |
| TPC        |  |  |  |  |  |       |          |          |
| Chl-a      |  |  |  |  |  |       |          |          |

Figure 1.5 Field Form FP5: Monthly Lake Cruise, Water quality, laboratory measurements.

### 1.4 Laboratory Analysis Methods

The WQ Component laboratory managers agreed already in 1966 to use the Standard Methods handbook 18<sup>th</sup> edition. The Consultant's review of the methods and parameters during the Inception Phase revealed that there were a few new parameters which required new or different methods. These were:

- Total nitrogen by distillation.
- Ammonia by the phenate method (Standard Methods 19<sup>th</sup> edition).
- Use of ethanol for chlorophyll determination.
- Silicate and total biogenic silicon methods to be developed.

These methods were introduced through in-laboratory training in Entebbe, Kisumu and Mwanza. More detailed training was given to Kisumu and Mwanza staff at DHI in Denmark, and followed up later during on-the-job training visits to the laboratories by the Consultant's specialists.

The conclusion is that the methods are well-known and agreed by the laboratories.

## **1.5 Training of Laboratory Managers and Staff**

Short training courses were organised by the Consultant for the laboratory managers and staff from Kisumu and Mwanza. The training was organised in three modules:

### *Module 1: Basic course for eutrophication laboratory analyses*

This training was at the DHI laboratories in Denmark, and involved two chemists/technicians from each country. The aim was to provide them with the necessary basic knowledge in analytical methods, practical QC procedures, basic QA management, laboratory safety and similar. A significant part of the training was carried out as practical laboratory training.

### *Module 2. On-site on-the-job training in eutrophication laboratory analyses*

This training was carried out by two experienced laboratory technicians from DHI on-site in Africa, during a one-month mission and a follow-up 3 week mission. The training covered all aspects of daily laboratory routines, and focussed on setting up the most needed analytical methods in the laboratories.

### *Module 3: Course in overall QA management for laboratory managers*

This module consisted of a two-week course in Quality Management for two laboratory managers, one from each laboratory. The module covered, among other things, the development of QA manuals as well as different aspects and techniques for internal and external quality control.

Reference is made to the final training report for more details.

The observations and recommendations of the Consultant's laboratory specialists were:

- The laboratory technicians (Kisumu and Mwanza) knew the analysis methods to use, but lacked confidence and practice. Long-term hands-on training is required.
- There is little internal sharing of knowledge and experience in the laboratories, eg. the techniques learnt by the staff trained in Denmark were not passed on to all the laboratory technicians.

## 1.6 Laboratory Manuals

DHI is the Reference Laboratory for Denmark for water analyses, and they have also specialised in establishing laboratories in developing countries and training the staff. Their experience has resulted in an optimal procedure and sequence of tasks. For example, when preparing manuals for the laboratory work, the "managerial" and "technical" aspects are always separated. The analytical methods at the technical level are always the most important in the start-up phase. When the methods are working reasonably well, it is time to get the proper QA work in place. There is naturally some overlap between the two phases. The Kisumu and Mwanza laboratories are still in the first "technical" phase.

The QA procedures related directly to the field and analysis work are a natural part of this technical phase such as field and laboratory forms, replicates, evaluation of blank values, internal control samples etc. and such procedures have been introduced in all three labs under LVEMP.

When the laboratory managers from Kenya and Tanzania were at the QA/QC training in Denmark, a start was made on writing QA/QC manuals for their respective laboratories. It is important to make them write the manuals themselves, based on well-established procedures but adjusted to the local conditions. QA/QC manuals coming directly from another source are rarely relevant or useful. It is important that they write down their analytical methods and procedures (based on Standard Methods) so that the staff can easily follow the instructions.

The QA/QC manuals consist of a combination of analytical methods and QA/QC procedures. The laboratory managers got rather far with the writing, but could not finish in the short period available.

An example of manual for an analytical method is included in the Attachment to this Chapter.

## 1.7 Data Validation

The working sessions revealed a myriad of errors in the data from the field and the laboratory, and data validation became a major part of the work at each session. Most of the errors relate to trivial mistakes and not, for example, to poor laboratory analyses. The trivial mistakes included formatting errors, typing errors, incorrect units, laboratory concentrations instead of conversion back to lake concentrations, repeated and omitted data in the database, wrong dates, arithmetic calculation errors, etc.

The more serious mistakes included:

- Incorrect field measurements, eg. temperature profiles with large depths of colder water overlying warmer water, water samplers releasing at incorrect depths.

- Incorrect storage of samples on-board the survey vessels.
- Contamination of reagents and samples in the laboratory.
- Incorrect post-analysis calculations which could not be traced back to lack of records.
- Wrong entries in the database, e.g. loss on ignition data for sediment traps occurred in the water quality database.

The Consultant's approach to the training of the WQ Component staff in data validation was concentrated on hands-on training and on the introduction of a number of data validation techniques at the working sessions.

### **Visual examination for "reasonableness"**

The first and most important technique is to visually examine the data (in numeric and graphical form) to see if the values are "reasonable", ie. what value range would one expect for each parameter in a sample collected at a given point in space and time. Initially, "generally known" value ranges for lakes were used, and as more data is collected, more specific values for Lake Victoria can be identified. This technique should be used on board the survey vessel to check profile data collected with a probe, as well as in the laboratory to check concentrations levels immediately after analysis. When applied at the time of analysis of the data as was done at the working sessions, there is no possibility of repeating the measurement and non-valid data will have to be discarded.

### **Statistical analysis**

The variability of the data has been checked with standard statistics calculated for all data (mean, medians, percentiles), drawing of "box" and "whisker" plots etc. The variability of each parameter was compared among the three laboratories and differences which could not be explained by natural causes were examined at the level of methodological problems.

The use of statistical methods may be extended when, a) the staff know what magnitude of values and vertical profile patterns to expect, and b) there are sufficient values to do a meaningful statistical analysis.

Regarding the amount of data, it should be realised that, at the time of reporting, there are a maximum of six profiles at any one station in the lake. The profiles are, by nature, very different in time and space, both horizontally and vertically, which limits the additional information given by a statistical analysis. The use of advanced statistical analysis will be more relevant when there are several years of monthly data and, even then, it will still be a delicate analysis because WQ data rarely follows a normal distribution - a fact that often affects the meaningfulness of classic parametric statistics (variance and standard deviation) and thereby classic methods such as ANOVA. '

### **Inter-parameter relations**

Data has been checked for outliers and for "normal" expected relations between parameters such as Chlorophyll-a/Secchi depth, organic N/organic P etc. "Suspect" data has been traced back to the laboratory for correction of possible errors. If reasonable corrections could not be made a decision was made as to whether the data should be discarded or kept in the operational database. Particular attention was paid to the training of the staff in the method at the working sessions.

A significant amount of data was discarded by the validation process and gave rise to the review of particular sampling and laboratory procedures.

The recommendation of the Consultant is that more training in field measurement techniques, and long-term training of the laboratory staff is required.

## **1.8 Inter-Laboratory Comparison**

As a part of the quality assurance programme an inter-laboratory experiment by the balanced uniform level method according to ISO 5725 -2 (1994) was carried out in December 2001. Three levels of spiked Lake Victoria water with "split levels" for two of the levels were used.

### **1.8.1 Accuracy – trueness and precision – of measurement methods and results.**

In all chemical analyses of materials there are errors consisting of errors at the laboratory – inappropriate methods, poor equipment, untrained people etc – and a random error occurring in every measurement under repeatability conditions. To estimate these errors with the purpose of minimising them, a collaborative inter-laboratory experiment on analyses of unknown samples can be performed. The results of such an experiment can be used to get a quantitative estimate of analytical quality within an area and to test the proficiency of each laboratory with the purpose of control and eventual accreditation. An inter-laboratory experiment shall be considered separate from the daily quality control (QC) carried out in the laboratories.

A design for inter-laboratory testing has been done in ISO/DIS 5725 and the requirements are: samples from  $q$  batches of materials, representing  $q$  different levels of the test, are sent to  $p$  laboratories which each perform exactly  $n$  replicative test results under repeatability conditions at each of the  $q$  levels – called a balanced uniform level experiment.

The test was carried out on 5 water quality parameters:

**PO<sub>4</sub>-P, NH<sub>4</sub>-N and NO<sub>3</sub>-N at levels: 0 - 0.5 mg/l**

**Total N and Total P at levels: 0 – 2 mg/l**

Each laboratory received 6 samples + 1 ampoule with test material for phosphate, ammonia and nitrate + 1 ampoule with test material for Tot N and Tot P + test certificates.

The samples were lake water from Lake Victoria nearshore Entebbe, some of which have been spiked with test material at different levels. The test material was provided by DHI, and only DHI knows the concentrations.

A test for homogeneity of the lake water sample by 10 replicate analyses was made by the Entebbe laboratory.

The samples were numbered 1 to 6 and "control sample" consisting of a diluted sample of test material.

The samples were delivered frozen and should be stored frozen ( $< -18^{\circ}\text{C}$ ). Analyses should be performed within 24 hours after thawing and adjustment to ambient temperature.

Analyses should be performed by the agreed methods with reagents from the normally used batches.

The analyses should be done by the laboratory personnel who normally do the specific analyses.

The results and a description of the method used should be reported on the form shown in Figure 1.6.

### **1.8.2 Preliminary results**

The results from Kisumu and Mwanza have arrived and scatter plots and regressions (Figure 1.7) have been performed on the Kenyan and Tanzanian results, but no Cochran and Grubb tests for outliers have been performed. The results from Uganda are not available due to procedural errors in the handling of samples.

The preliminary conclusion is that there is still a need for an improvement in both analytical precision and accuracy. The conclusion reinforces the recommendation for long-term training.

**Name of Laboratory:**.....

**Sample Nr**

| Parameter               | 1 | 2 | 3 | 4 | 5 | 6 | control |
|-------------------------|---|---|---|---|---|---|---------|
| NH <sub>4</sub> -N mg/l |   |   |   |   |   |   |         |
| NO <sub>3</sub> -N mg/l |   |   |   |   |   |   |         |
| PO <sub>4</sub> -P mg/l |   |   |   |   |   |   |         |
| Tot N mg/l              |   |   |   |   |   |   |         |
| Tot P mg/l              |   |   |   |   |   |   |         |

**Methods used:**

**NH<sub>4</sub>-N:**

**NO<sub>3</sub>-N:**

**PO<sub>4</sub>-P:**

**Tot N:**

**Tot P:**

**Date and signature:**.....

Figure 1.6 Form for reporting of analysis results from inter-laboratory comparison.

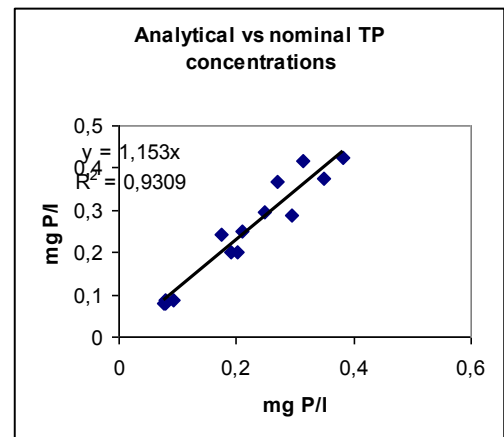
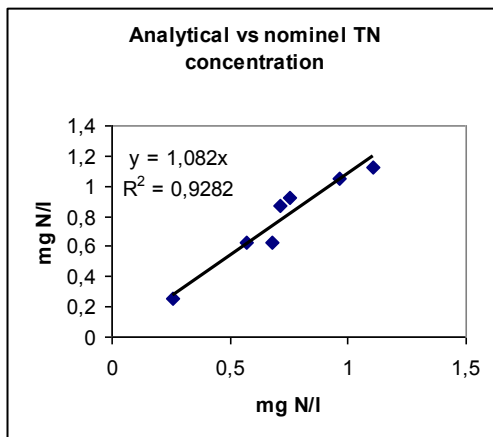
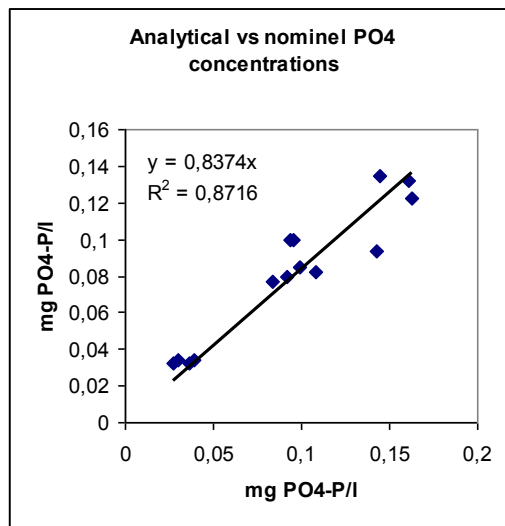
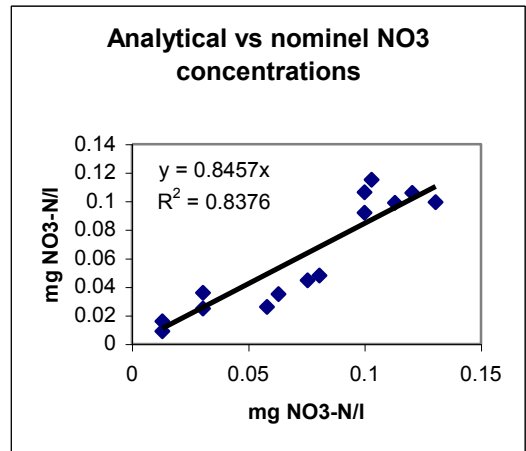
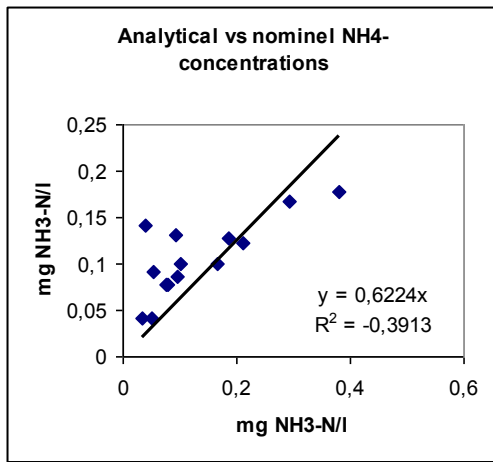


Figure 1.7 Analytical vs nominal concentrations



## **1.9 Recommendations**

The Consultant strongly recommends the inclusion of additional training in the bridging phase and in Phase II of LVEMP. Training should cover both the field and the laboratory.

### **Field measurement techniques**

The field staff require training in actual field techniques with the various instruments and the immediate, on-board analysis of data to reveal measurements errors and the need for repetition of the profiles and sampling. Training on three lake cruises is expected to be sufficient to build up the necessary routine.

### **Laboratory methods and QA/QC**

Long-term, hands-on training of the laboratory staff is required, particularly in Kisumu and Mwanza. It is a well-established fact that it takes years to bring a new laboratory up to the stage where it consistently produces quality analyses - where the technicians know all that can go wrong and how to detect errors, interferences due to sample composition, possible contaminations, etc. This process has only just started in Kisumu and Mwanza, and Kisumu doesn't even have proper laboratory facilities yet. The staff need continuous on-the-job training and practice to build up confidence and routine before they can produce quality data which can stand up against the rigorous requirements of Reference Laboratories. and intra- and inter-lab calibrations.

The laboratory training should continue over at least 2 years, with visits by specialists for 3 to 4 months each year. The training should include not only analysis methods and post-processing of data, but also completion of the methods and QA/QC manuals.



## **Attachment to Chapter 12**

**Example from Analysis Methods Manual for Mwanza laboratory**



