QUALITY ASSURANCE AND QUALITY CONTROL DOCUMENT FOR LAKE MARIOUT STUDY

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INTRODUCTION AND PROJECT DESCRIPTION

1.1 INTRODUCTION

This document outlines the chemical, physical, and biological data quality assurance standards by which the sampling and analysis efforts shall be conducted at the Laboratories of the High Institute of Public Health, Environmental Health Dept.

This document presents the detailed site-specific field and laboratory procedures to be followed, to insure the quality and integrity of the sampling and the analytical procedures adopted during the conduct of the Lake Mariout Study. The data will be further used in the environmental impact assessment of the treated waste discharge to the Lake, which will be the final recipient of the primary treated combined industrial and domestic wastewater effluent.

This document is prepared to assure that the collection and analysis of the samples are both performed in the highest quality manner and the results will closely represent the status of pollution in the Lake. Sediment and water samples will be collected and preserved according to the EPA standards of sampling and preservation. Sample analysis will be conducted according to Standard Methods of Water and Wastewater Analysis.

1.2 SITE LOCATION AND DESCRIPTION

Lake Mariout is a brackish water lake receiving its water from agricultural drains, which collect drainage water from the Delta region and flow by gravity to El-Omoum Drain. Omoum Drain, in turn, discharges to the south-west corner of the Lake's main basin, the basin of concern in this study. The second drain is El-Kalaa Drain which discharges at the south-east corner of the basin under study. In addition, Noubaria Canal is considered the fresh water source for the Lake, yet its water by the time it reaches the Lake is saline and polluted. Industrial waste effluents are also mixed with domestic effluents and are discharged into the north side of the Lake at Moharrem Bey, at Ghiet El Enab, and at Karmous.

During Phase I, the East Treatment Plant, after primary treating 410 ML/D, will discharge its effluent into an agricultural drain leading to El-Kalaa Drain and finally into Lake Mariout. The West Treatment plant, after primary treatment of the combined domestic and industrial effluents, will discharge 175 ML/D into the north-west corner of the Lake. According to the Alexandria Master Plan, the three north sewage industrial outfalls will be diverted to the collection system, and their discharges will then flow to the West Treatment Plant.

The lake site is about 3000 feddans, with a very shallow bottom reaching 150 cm at its deepest location and a depth of 50 cm at shore locations around the lake periphery.
The east side of the basin is presently being filled with solid wastes and garbage collected from the City. Solid waste leachate from this side is an added nonpoint source of pollutional load to the Lake basin.

Excessive weed growth around the periphery and on isolated small islands in the Lake hinders the process of natural reaeration in the basin. The excessive organic and toxic chemical loads reaching, the basin in addition to low natural reaeration rate, contribute severely to the lake's continuous anaerobic state. The lake water level is maintained at a relatively constant level by means of the EL–Max pumping station where water is discharged through a dug canal to the Mediterranean Sea.

1.3 SITE BACKGROUND

Lake Mariout used to be a highly productive fishing lake as well as a recreational lake for wild duck hunting. The lake was divided into four basins upon the construction of the highway connecting Alexandria with the desert areas around it and with Cairo. As the area around the lake became more developed, the cleanest east basin, used as a fishery basin, was filled with garbage and used as a garden. The eastern side of the main basin is in the process of also being filled with domestic and industrial solid wastes.

Industrial development has occupied a relatively wide stretch around the lake with various industrial activities dumping their untreated wastes directly into the Lake. Domestic and industrial wastes generated and collected in the sewer system in the eastern part of the Governorate are also discharged to the Lake without any treatment either.

Domestic sewage, mixed with agricultural drainage water, find their way to the main basin of the Lake through Gheit El Enab and Karmous drains. At the farthest southwest corner of the main basin, petroleum companies discharge their cooling and processing water, which is laden of oils and petroleum derivatives.

1.4 PROJECT OBJECTIVES

Lake Mariout monitoring has been carried several times during the past years. Since the polutional status of the lake is ever escalating, it was determined by USAID that an evaluation was now needed of the main basin condition, ahead of its use as the final recipient of the primary treated combined industrial and domestic effluents, discharged by the East and West treatment Plants. Any environmental adverse impacts, and any possible remedial alternatives, could be determined through the generation of data collected during this study.

The major part of the present land base sources of pollution will be collected in the sewer system and diverted to the two treatment plants for primary treatment and
discharged to the lake at two points. The effect of this waste concentration can be evaluated if the background status of the Lake's main basin is documented. Any remedial actions needed can be assessed.

Biological analyses of the lake water samples will give a clear indication of the Lake's self-cleaning capabilities. To achieve this, water samples for chemical, bacterial and algal analyses will be collected in proper containers with preservatives to maintain the samples in their original form. Heavy metal analyses will also be performed on these samples.

Sediment samples will be collected for analyses of their heavy metals, as well as their organic and inorganic constituents. Water samples from the land base sources will be collected and analyzed for their physical and physiochemical parameters in addition to their heavy metal contents. An attempt will be made to draw contour lines for the different pollutants in the Lake's main basin, using the computer graphic capabilities of WWCG. Correlation of meteorological data with the prevailing ecological conditions of the lake will be studied.

1.5 SAMPLING SCHEDULE

A reconnaissance survey of the Lake's, land base sources of pollution, as well as Kalaa and Smouha Drains, was started in March 1992. The sampling schedule will be repeated for 12 months to cover seasonal variations encountered in the Lake. Sampling will be carried out on two consecutive days. During the first day, samples from drains and landbase outfalls will be collected. On the second day, water and bottom sediments samples from the main basin will be gathered and sent to the laboratory within half an hour from sampling termination. Usually, lake sampling takes about 1.5–2 hours. A similar time is expected for land base samples.

1.6 DATA USAGE

The organic and inorganic analyses of the water and bottom sediments in the Lake will provide background information, which will be compared with the similar estimated parameters for the Lake after receiving the Phase I wastewater effluents. This will allow for identification of important factors affecting the self-purification process of the Lake. Bottom sediment analyses will allow the estimation of the organic and heavy metal loads that may be released from the bottom to the Lake, and which would add to Lake's total pollution loading.
2. QUALITY ASSURANCE OBJECTIVE

2.1 GENERAL

The comprehensive Quality Assurance (QA) objectives for the Lake Mariout study is to provide guidelines for all field and laboratory procedures to be followed in this study and in any future surveys of the main basin of the Lake in order to assess the Phase I impacts on the Lake.

The intention of the sampling and analysis effort is to produce data of acceptable quality to allow for an accurate evaluation of the chemical, biological, and ecological impacts of the present and future sources of pollution. An additional effort will be to provide a basis for any remedial restoration of the Lake in order to accommodate the future discharges.

The main QA objective for this study is to obtain all measurements as representable as possible for the actual site conditions and all data resulting from sampling and analysis activities to be comparable. The use of accepted, published, sampling and analysis methods, as well as the use of standardized units, shall assure the comparability of the data.

The major characteristics of data quality to be addressed, during development and presentation of the sampling and analytical plan, are defined hereinafter.

2.2 ACCURACY

Accuracy is defined as the degree of agreement of a measurement (or measurement average) with an accepted reference or true value. It is a measure of system bias and is usually expressed as a percentage of the true value.

Accuracy will be determined in the laboratory through the use of spike and duplicate analysis. The head team shall select a spike sample every five samples to be analyzed and measured following the same parameter scheme under investigation. A frozen volume of each sample will be preserved till the finalization of the monthly analysis program, in case any sample needs to be rechecked. In this way, a fresh sample can be obtained upon thawing. Algal analysis will be maintained by preserving the samples with iodine solution and refrigeration, as freezing can alter its quality. Spiked samples of known chemical concentration will be prepared under the direct supervision of the team leader by another lab chemist not involved with the study and without revealing the name of chemicals being used, just referring to them as chemical A, B etc.

Enough volume of both water (Liters) and sediment samples (500 C.C) will be collected at each sampling station in the Lake basin. Sterile glass bottles will be used
for bacterial analysis, and will be sent to the laboratory in an ice chest surrounded with plastic bags filled with ice.

Sampling accuracy shall be maintained by adhering strictly to the procedural protocol agreed upon by the team and WWCG, in order to achieve the goals of the study.

2.3 PRECISION

Precision is a measure of the amount agreement among individual measurements of the same parameter under similar conditions. It is expressed in terms of relative percent difference (RPD) between replicates or in terms of the standard deviation when three or more replicate analyses are performed.

Precision shall be determined through the use of duplicate analyses for the same sample. The RPD between the two results shall be calculated as a measure of analytical precision.

2.5 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another. To achieve comparability in this study, the data generated will be reported using units of ug/l, ug/kg and mg/kg. Securely prepared standard reference samples will be locally prepared to ensure data validation.

2.6 PROCEDURES FOR DATA ASSESSMENT

The precision values will be presented in a table showing the variability for replicate measurements of the same parameter, and are expressed in terms of relative percent difference for duplicate measurements made on samples. Accuracy values include components of both random error (i.e., variability due to imprecision and systematic error, (i.e., bias), and thus reflect the total error for a given measurement.
3.0 SAMPLING

3.1 SAMPLING OBJECTIVES

WWCG will conduct the survey of the Lake's main basin, its contributing drains, and the sewer outfalls discharging to the Lake. A representative sample of the water column and the bottom sediment at each Lake site will be collected using a bottom sampler and a BOD sampler at three levels identified at 30 cm from the bottom, middle depth, and 30 cm below the water surface. The three samples will be equally mixed for each site sample.

For the drain sampling, water samples will be collected using the previous technique, to give a clear indication about the current status of the drains pollution load and to assess the impact of primary treated wastewater effluent discharged by these drains. The effect of the industrial effluents discharged from Moharrem Bey Industrial Complex will be also assessed by the sampling the outfalls ahead of their discharge into the Lake.

3.2 SAMPLING LOCATION

Attached Figure i shows the selected sample locations in the Lake's main basin and also the presently contributing land base sources of wastewater. Note that some previously selected sample locations have been canceled to minimize the total number of sampling sites. Overall, seventeen sampling sites are required as described below:

Site No. 1: Represented by Sample No. 3. This sample represents the quality of the water generated as a result of mixing agriculture drainage water flowing in El Qualaa Drain, with the domestic wastewater discharged from the ETP, from there to the Hydrodrome Drain and then pumped by Qualaa Pumping Station.

Site No. 2: Represented by Sample No. 5. This sample demonstrates the water quality at the end of EL Qualaa Drain ahead of its mixing with the lake water. This sample will give an indication the load on the main lake basin in order to assess the self-purification capacity of the drain, now and in the future, after the Phase I project is implemented. The water sample will be collected from over the existing highway bridge at this location.

Site No. 3: Represented by Sample No. 6. This sample represents the combined industrial and domestic wastes pumped by Moharrem Bey pumping station to the northeast corner of the main basin, directly ahead of its mixing with the lakewater. The Phase I system will divert this point source to the West tunnel for treatment at the West Treatment Plant. The effluent of this outfall is a mixture of raw industrial wastewater generated by a wide variety of industries namely, pulp and paper, edible oil processing, soap and glycerol processing, match industry, textile industry, yeast processing, starch and glucose industry, and small scale confectionery plants.
Site No. 4: Represented by Sample No. 7. This sample represents the combined domestic, industrial and minor agricultural drain water after it mixes with lakewater at the north side of the basin. This site in previous surveys was referred to as Gheit ElEnab drain. The water quality in the Drain has changed to combined domestic waste due to the urbanization of the area south of the Mahmoudia Canal. This outfall will be diverted to the west tunnel running parallel to Mahmoudia Canal as part of Phase I.

Site No. 5: Represented by Sample No. 8. This sample describes the effluent discharged from Forn ElGeraya outfall. This outfall used to be basically agricultural drain water. However, due to the surrounding area becoming both residential and industrial area without any planned sewer facilities, these wastes are now discharged to the Lake at that location. This outfall will be eliminated upon the implementation of Phase I.

Site No. 6: Represented by Sample No. 9. This sample is taken from the discharge of Elmetras, also the outfall for Pumping Station 3 West. This outfall will be also diverted to the West Treatment Plant as part of Phase I.

Site No. 7: Represented by Sample No. 20. This sample is intended to demonstrate the water quality of the Lake’s basin as pumped by El Max Pumping Station to the Sea. Data collected at this site will also help determine the final pollutional load discharged from all land based sources which impact the sea at El Max Bay.

Site No. 8: Represented by Sample No. 24. Data collected at this site demonstrate the lake water quality as impacted by the discharge of the petroleum refineries located to the south. This site is also affected by the Noubaria canal, which is a higher salinity water canal than the similar fresh water canals. El Omoum Drain is another added source of agricultural wastewater mixed with domestic wastes generated by the villages close to its borders.

Site No. 9: Represented by Sample No. 19. Data collected at this site gives an indication of the pollutional load contributed by this collective agricultural drain.

Site No. 10: Represented by Sample No. 22. This site is located at what used to be the cleanest corner of the Lake, as it is the least affected by pollution discharged at the north and south borders of the main basin.

Site No. 11: Represented by Sample No. 21. This location is an intermediate location at the center of the basin. It is chosen to demonstrate the resultant water quality as affected by all sources of pollution in the Lake.

Site No. 12: Represented by sample No. 16. Data collected at this location will indicate the impacts of domestic wastewater discharge from El Kalaa Drain after mixing with the lakewater. The effect of water stagnation at this corner of the Lake can therefore be assessed.
**Site No. 13**: Represented by Sample No. 10. This sample represents the lake water 500 meter from the east border of the basin. This station is impacted by the decomposition products and leachates from the City's landfilling operation taking place along the east side of the main basin. Heavy plants and weeds are making sampling close to the dumping site unachievable. Accordingly, it was decided to sample the lake water at a distant about 50 meters west of the Lake’s east bank.

**Site No. 14**: Represented by Sample No. 11. Samples at this site will represent the pollutational effects of the industrial wastes discharged from Moharrem Bey Industrial Complex, mixed with the domestic waste of the residential area close to the industrial area. This site is also affected by Gheit El Enab Drain.

**Site No. 15**: Represented by Sample No. 23. This sample demonstrates the pollutational status of the middle north sector of the Lake, as impacted by stagnation and the two closer outfalls, namely Gheit El Enab and El Metrass outfalls.

**Site No. 16**: Represented by Sample No. 12. Samples at this site are collected about 500 meters south to the North border of the Lake in front of Forn El Geraya outfall.

**Site No. 17**: Represented by Sample No. 13. The lakewater in this site is affected by the discharge of El Metrass Pumping Station. Pollution status at this site is controlled by the prevailing winds, which push the flow superficially in their direction. The area around this site is relatively clear of the excessive weed growth.

### 3.4 FIELD QUALITY CONTROL FOR SAMPLING

To assure a true presentation of the water quality sampled, water samples will be collected from three depths at each site, and will be further mixed in a plastic container, clearly marked with a non-flowing black marker on both sides of the container. Only one container will be used for each same sampling site. Bottom samples will be collected by mixing three bottom catches from the bottom sampler and then taking one quarter of the total amount at random.

Temperature readings will be taken as the average of two thermometer readings measured at each site. Depth will be measured twice and averaged at each site. Dissolved Oxygen is measured at the laboratory after fixing it at the sampling site. The BOD bottle will be filled with minimal disturbance at the three depths at each site. Samples from land base polluted water sources will be collected at a middle point of the source, with the BOD sampler to achieve proper mixing of the sampled water at all depths. To collect the volume of at least 12 liters from each site, the BOD sampler will be filled at each site at least 11 times and emptied in the container, which will result in good sample representation of the water at each location.

Bacteriological samples will be collected in sterilized glass bottles from the water samples collected at each site.
Disturbance effect of the water in the lake, due to the mixing action of the boat propeller will be minimized by shutting down the motor 50 meters away from the sampling location and allowing the boat to move under its own inertia towards the sampling site.

3.5 SAMPLE CONTAINERS AND PRESERVATION

Table (1) lists the type of containers and sample preservatives required, and the maximum holding time allotted for each analysis. Plastic containers were purchased from a local supplier from the type used for storage of drinking water. Thorough washing will be carried on each container after and ahead of its use, with hot water and a pure liquid detergent concentrate free of phosphates. Further rinsing with cold water for three times with at least two liters of tap water followed by 500 ml distilled water will be part of the sample container preparation.

Glass containers will be of good quality glass for water sample collection for biological and bacteriological analysis. Bottles with narrow mouth and a capacity of 250 ml will be used after autoclaving for the bacterial analysis.

Acid digested water samples and bottom sediments will be sent to the heavy metals analysis laboratory in 50 ml capacity medical grade tight plastic containers labeled with the sample no. and type of sample whether water or bottom sediment.

Samples collected for biological analysis will be taken in high quality dark glass to minimize the light effect on algal growth. The collected sample from variable depths will be stored in these bottles at each site.

3.6 ANALYTICAL PROCEDURES

Table (2) summarizes the analytical parameters measured and the analytical techniques to be used during this study. All analysis will be carried according to the Standard Methods of Water And Wastewater Analysis published 1987.
TABLE (1)

SAMPLING AND PRESERVATION
OF SAMPLES ACCORDING TO MEASUREMENT

<table>
<thead>
<tr>
<th>Measurement</th>
<th>Vol. Req. (ml)</th>
<th>Container</th>
<th>Preservative</th>
<th>Holding Time (6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidity</td>
<td>100</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>100</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>BOD</td>
<td>1000</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>6 Hrs.</td>
</tr>
<tr>
<td>COD</td>
<td>50</td>
<td>P, G</td>
<td>H₂SO₄ to pH&lt;2</td>
<td>7 Days</td>
</tr>
<tr>
<td>Chloride</td>
<td>50</td>
<td>P, G</td>
<td>None Req.</td>
<td>7 Days</td>
</tr>
<tr>
<td>Color</td>
<td>50</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Dissolved Oxygen Probe</td>
<td>300</td>
<td>G only</td>
<td>Det. on site</td>
<td>No Holding</td>
</tr>
<tr>
<td>Winkler</td>
<td>300</td>
<td>G only</td>
<td>Fix on site</td>
<td>No Holding</td>
</tr>
<tr>
<td>Hardness</td>
<td>100</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 Days</td>
</tr>
<tr>
<td>Metals Dissolved</td>
<td>200</td>
<td>P, G</td>
<td>Filter on site HNO₃ to pH&lt;2</td>
<td>6 Mos.</td>
</tr>
<tr>
<td>Suspended</td>
<td></td>
<td></td>
<td>Filter on site</td>
<td>6 Mos.</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>P, G</td>
<td>HNO₃ to pH&lt;2</td>
<td>6 Mos.</td>
</tr>
<tr>
<td>Nitrogen</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ammonia</td>
<td>400</td>
<td>P, G</td>
<td>Cool, 4°C H₂SO₄ to pH&lt;2</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Kjeldahl</td>
<td>500</td>
<td>P, G</td>
<td>Cool, 4°C H₂SO₄ to pH&lt;2</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Nitrate</td>
<td>100</td>
<td>P, G</td>
<td>Cool, 4°C H₂SO₄ to pH&lt;2</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Nitrite</td>
<td>50</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Oil &amp; Grease</td>
<td>1000</td>
<td>G only</td>
<td>Cool, 4°C H₂SO₄ to pH&lt;2</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Measurement</td>
<td>Vol. Req. (ml)</td>
<td>Container</td>
<td>Preservative</td>
<td>Holding Time (6)</td>
</tr>
<tr>
<td>---------------------------</td>
<td>----------------</td>
<td>-----------</td>
<td>-----------------------------------</td>
<td>------------------</td>
</tr>
<tr>
<td>PH</td>
<td>25</td>
<td>P, G</td>
<td>Cool, 4°C Det. cn site</td>
<td>6 Hrs.</td>
</tr>
<tr>
<td>Phosphorus Orthophosphate, Dissolved</td>
<td>50</td>
<td>P, G</td>
<td>Filter on site Cool, 4°C</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Sulfide</td>
<td>50</td>
<td>P, G</td>
<td>2 ml zinc acetate</td>
<td>24 Hrs.</td>
</tr>
<tr>
<td>Temperature</td>
<td>1000</td>
<td>P, G</td>
<td>Det. on site</td>
<td>No Holding</td>
</tr>
<tr>
<td>Turbidity</td>
<td>100</td>
<td>P, G</td>
<td>Cool, 4°C</td>
<td>7 Days</td>
</tr>
</tbody>
</table>
4.0 SAMPLE CUSTODY

4.1 GENERAL

Special consideration has been given for the validation of the environmental measurement data to demonstrate that samples have been obtained from the location stated and that they have reached the laboratory without alteration.

The field team leader is responsible for overseeing and supervising the implementation of proper sample delivery at the laboratory, as well as securing them in the lab in a restricted area where nobody other than the authorized personnel can deal with them. Samples while not in use will be kept in refrigerators in order to minimize any possible changes in their water quality.

Once the samples arrive at the lab, a volume of 500 ml is kept frozen in the deep freezing section of the lab refrigerators to be thawed and analyzed in case the data for any physical or chemical parameter needs to be rechecked. The team leader will assure this step is carried out upon sample arrival at the laboratory.

4.2 LABORATORY SAMPLE CUSTODY PROCEDURE

Upon sample arrival, the chief chemist is responsible for accepting the samples and checking with the team leader to insure that the sample numbers, locations, and tags are all accounted for in case any sample is damaged during shipment and transportation.

Chief chemist must verify that samples are preserved and collected in proper containers. In case of mistakes, immediate corrective action is required. The chief chemist will also record the general information of the samples, including the Project name, sample number, date of sampling and source of samples, in a log-in book.

Project supervisor is responsible for the different analytical task appointments within the specialized staff. Chemical and physiochemical analysis are carried by a group of five analysts to insure accurate results. Each analyst has been assigned certain parameters in order to minimize variations in the results due to personal variability.

Bottom sediment analysis will be carried out by one expert during the project duration. Bacterial and biological analysis will also be performed by one specialist. Heavy metals analysis for both water and bottom sediments will be acid digested and analyzed by atomic absorption spectrophotometry by another specialist.

Overall supervision on the samples is the responsibility of the chief chemist, double checked by the project supervisor.
BOD bottles will be kept in a wooden rack which holds 30 bottles at one time. Bacterial analysis bottles will also be transported in wooden racks to prevent their spillage. Same procedures will be maintained for the biological samples using a wider rack with smaller number. Special racks will be used for keeping the DO fixing solutions, thermometers, and preservative solutions, to prevent breakage or spillage.

4.3 PHOTOGRAPHS

The team leader will document, through the use of color photography, various on-site environmental conditions of the different sampling locations as deemed necessary. Examples of items that may require such photographic documentation include:

- General site pollutional conditions.
- Exact sample locations.
- Physical appearance of environmental samples.

4.4 FIELD LOGBOOKS

A field logbook will be maintained by the field team leader and a record of each sample site, depth, and water temperature will be recorded. Observations regarding the visible status of pollution in the lake sites will also be recorded.

The field team leader has the responsibility of emphasizing the logbook completeness and accuracy. Entries into the logbook shall include:

- Weather conditions, sampling date and time.
- pH, water clarity, and temperature.
- Description of the sampling sites landmarks.
- List of any changes from standard operation procedures, decisions made in the field, and other pertinent information.
- Comments relative to any problem areas that occurred during the day’s activities, their final resolution, and any anticipated impact on the outcome of the field investigation.
5.0 CALIBRATION PROCEDURES AND FREQUENCIES OF ANALYTICAL INSTRUMENTS

5.1 LABORATORY INSTRUMENTATION

Instruments used in the laboratories of the High Institute of Public Health, Department of Environmental Health are all subject to continuous calibration before they are used. The manner in which various instruments are calibrated is dependent on the particular type of instrument and its intended use.

All sample measurements are made within the calibrated range of the instrument. Preparation of all reference materials will be documented in a standards preparation notebook.

Instrument calibration typically consists of two types:

- Initial calibration
- Continuing calibration.

Initial calibration procedures establish the calibration range of the instrument and determine the instrument response over that range. Usually the calibration is carried out with three concentrations, having low, medium and high concentrations. The instrument response over the range is absorbance, or transition, which can be expressed as a linear model with a response factor, or as an amount vs. response plot.

Continuing calibration may be used within an analytical sequence to verify stable calibration throughout the sequence and/or to demonstrate that the instrument response did not drift during a period of non-use of the instrument. Calibration is further ensured by the periodic analysis of quality control samples during the course of instrumental analysis of field collected samples.

The following analytical instruments shall be used to analyze samples according to The Standard Methods For Examination Of Water And Wastewater, the fourteenth Edition, and EPA Methods For Chemical Analysis Of Water and Wastes.

- pH meter
- Conductivity meter
- Spectrophotometer
- Atomic absorption Spectrophotometer
5.2 FIELD TEST EQUIPMENT

Prior to the use of any field test equipment, proper calibration shall be ensured. Specific calibration of certain instruments is described as follows:

pH CALIBRATION

A field digital pH meter shall be used throughout this investigation in order to determine the pH of various water samples. All pH buffer solutions used to calibrate the pH meter are purchased from Fisher Scientific Co. USA.

DO FIXATION

A BOD bottle will be filled with the composite water sample from the three depths collected by the DO sampler. Fixation using manganous sulphate solution and alkaline azide solution will be done immediately after the sample collection in the field.
6. ANALTICAL PROCEDURES

6.1 GENERAL

The analytical methods specified for the collected samples during this study are summarized in the following table:

### TABLE (2) ANALYTICAL PARAMETERS AND PROCEDURES IN LAKE MARIOUT STUDY

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>ANALYTICAL PROCEDURE</th>
<th>UNITS OF MEASUREMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>Glass Thermometer</td>
<td>Degrees Centigrade</td>
</tr>
<tr>
<td>Conductivity</td>
<td>Conductivity meter</td>
<td>umhos/cm</td>
</tr>
<tr>
<td>Total Solids</td>
<td>Drying at 103-105°C</td>
<td>mg/L</td>
</tr>
<tr>
<td>Dissolved Solids</td>
<td>Filtrable Solids dried at 103-105°C</td>
<td>mg/L</td>
</tr>
<tr>
<td>Volatile and Fixed Solids in Water Samples</td>
<td>Ignition at 550°C</td>
<td>mg/L</td>
</tr>
<tr>
<td>Volatile and Fixed Solids in Solid and Bottom Sediments</td>
<td>Ignition of dry weight at 550°C</td>
<td>mg/L</td>
</tr>
<tr>
<td>Heavy Metals</td>
<td>Acid Digestion Followed by Atomic Absorption</td>
<td>mg/L</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>Titrimetric analysis</td>
<td>mg/L CaCO₃</td>
</tr>
<tr>
<td>Chlorides</td>
<td>Argentometric Titration</td>
<td>mg/L Cl⁻</td>
</tr>
<tr>
<td>Hardness Total</td>
<td>EDTA Titration</td>
<td>mg/L CaCO₃</td>
</tr>
<tr>
<td>Calcium Hardness</td>
<td>EDTA Titration Using Murexide Indicator</td>
<td>mg/L CaCO₃</td>
</tr>
<tr>
<td>Magnesium Hardness</td>
<td>By Difference</td>
<td>mg/L CaCO₃</td>
</tr>
<tr>
<td>Ammonia (Nitrogen)</td>
<td>Distillation Followed by Nesslerization</td>
<td>mg/L NH₄</td>
</tr>
<tr>
<td>Nitrate (Nitrogen)</td>
<td>Chronotropic Acid calorimetric determination</td>
<td>mg/L NO₃</td>
</tr>
<tr>
<td>Nitrate (Nitrogen)</td>
<td>Deoxidization Method</td>
<td>mg/L NO₂</td>
</tr>
<tr>
<td>PARAMETER</td>
<td>ANALYTICAL PROCEDURE</td>
<td>UNITS OF MEASUREMENTS</td>
</tr>
<tr>
<td>------------------------</td>
<td>--------------------------------------------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>Dissolved Oxygen</td>
<td>Audiometric Method <em>Azide Modification</em></td>
<td>mg/L O₂</td>
</tr>
<tr>
<td>pH</td>
<td>Glass electrode</td>
<td></td>
</tr>
<tr>
<td>Phosphates</td>
<td>Stannous Chloride Method</td>
<td>mg/L P&lt;sub&gt;0&lt;/sub&gt;</td>
</tr>
<tr>
<td>Sulfates</td>
<td>Turbidimetric Method after filtration</td>
<td>mg/L S&lt;sub&gt;0&lt;/sub&gt;</td>
</tr>
<tr>
<td>Grease &amp; Oil</td>
<td>Wet Extraction by Petroleum Ether after Acidification</td>
<td>mg/L</td>
</tr>
<tr>
<td>Oxygen Demand</td>
<td>Winkler Modified Azide</td>
<td>mg/L O₂</td>
</tr>
<tr>
<td>Biochemical BOD</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oxygen Demand Chemical COD</td>
<td></td>
<td>mg/L O₂</td>
</tr>
</tbody>
</table>

### 6.2 LABORATORY STANDARDS AND REAGENTS

Laboratory standards and reagents are obtained from a local supplier who imports them from Fisher And Coli Parmer Chemicals in Europe. They are all Analytical Grade.

All standards and laboratory reagents and common laboratory solvents are dated upon receipt. The preparation and use of all standards are recorded in bound laboratory notebooks to document their traceability.

Additional information recorded includes the date of preparation, concentration of the prepared solution, and name of preparer.

### 6.3 LABORATORY GENERATED QC SAMPLES

#### 6.3.1 Calibrated Check Sample

One calibration check sample (CCS) shall be analyzed for every 10 samples analyzed sequentially for all project–specified parameters. A CCS is chosen as one of the mid-range working calibration standards that is reanalyzed periodically throughout the sample analysis to verify that the original calibration is still valid. A composite CCS of known concentrations of chlorides, sulfates, phosphates, and nitrates will be analyzed to determine the recovery of each parameter in the presence of the others (spiked Sample).
6.3.2 Method/Reagent Blank

One method/reagent blank shall be analyzed with each sample batch tested. A method blank is comprised of laboratory-pure, analyte-free water carried through the entire sample preparation and analysis procedure. Analysis of the method blank provides a check of the background contamination due to sample preparation procedures.

6.3.3 Laboratory Replicates

One sample per monthly batch of samples will be analyzed in replicate. A replicate sample is produced by dividing a single collected sample into two equal parts for the purpose of determining analytical precision.

6.3.4 Matrix Spike /Matrix Spike Duplicates

A spiked sample analysis will be performed with each parameter determination, using a multi-chemical spiked sample. If the recovery is not within the acceptable criteria limits as specified in the Standard Methods for Water and Wastewater Analysis, the data of those samples and the spike must be repeated.

6.4 BIOLOGICAL ANALYSIS

Water samples collected from the three depths of the Lake, as well as those collected from land base sources, will be kept in wide mouth glass bottles in ice chests and after the addition of preservative solution. All water samples will be concentrated by means of Schedwick rafter funnel. The concentrated volume is counted under the microscope using the standard Schedwick cell for algal species identification, and counting. All counts will be presented as counts/liter.

6.5 BACTERIAL ANALYSIS

The collected samples for bacterial analysis will be subject to serial dilution with sterile water to get the proper dilution of samples that can be cultured and counted on the agar plates.

The MPN technique will be followed to determine the fecal coliform bacterial counts. The presumptive test will confirm the presence of the fecal coliform bacteria. All data will be presented as counts/100 ml.
6.6 DATA PRESENTATION

Data of all sample analysis will be presented in monthly cumulative data tables. Each parameter will be presented in a separate table over the different sites for the consecutive 13 months of sampling.
LEGEND:

- MAJOR OUTFALLS
- PUMP STATIONS
- FINAL SAMPLING LOCATIONS
- DELETED SAMPLING LOCATIONS

FIGURE 1
SAMPLE NUMBERS FOR LAKE MARYOUT MONITORING PROGRAMS