

APPENDIX-2

STANDARD OPERATING PROCEDURES OF WATER QUALITY ANALYSIS

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1 Determination of pH

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN6492:1999 (ISO10523:1994) Water quality - Determination of pH

Type of sample and Range

- This method is applicable to all types of water and waste water samples in the range from pH 3 to pH 10.

Interferences

- The temperature, some gases and organic materials interfere with the pH-measurement.
- Suspended materials in the sample may cause significant errors (suspension effect). Wait for sedimentation and only insert the electrodes in the clear fraction.
- When measuring sewage and some surface waters, there is a particularly high risk of smearing the electrodes or contaminating the membranes and diaphragms with oil, grease or other contaminants.

II. PRINCIPLE

- Definition of pH is as follows.

pH is defined as the decimal logarithm of the reciprocal of the hydrogen ion activity, a_{H^+} , in a solution.

$$pH = -\log_{10}(a_{H^+}) = \log_{10}\left(\frac{1}{a_{H^+}}\right)$$

- There are three different methods of pH measurement: pH indicator paper, liquid colorimetric indicators and electronic meters.

III. SAMPLING AND SAMPLE PRESERVATION

- Determination of the pH of water should, if possible, be made in situ. If this is not possible, for example with well water or when access to a lake or river is very difficult, the measurement should be made immediately after the sample has been obtained.

IV. APPARATUS

- 1) Beaker 100ml
- 2) Tissue paper
- 3) pH meter.

V. REAGENTS

- 1) Buffer solutions: pH 4.0, 7.0 and 10.0
- 2) Pure water

VI. PROCEDURE

Calibration

This is an example of the calibration. Please follow the manual. The calibration of pH meter shall be carried out each day of pH tests.

- 1) Switch on meter

- 2) Fill the standard solution in the container
- 3) Wash the sensor module with the water and wipe with tissue
- 4) Put the sensor module in the container filled with pH 7 standard solution and stir the container three times.
- 5) Leave it until the pH indication is stabilized and press CAL/ENT key
- 6) When the pH is stabilized, a beep sounds again and the calibration mark 7 is displayed
- 7) Take out the sensor module from the calibration container, wash it with the water and wipe the water off with tissue.
- 8) Put the sensor module in the calibration container filled with pH 4 standard solution and repeat the same procedure with above e) to g).
- 9) Put the sensor module in the calibration container filled with pH 10 standard solution and repeat the same procedure with above e) to g).
- 10) After having finished the calibration, wash sensor module with pure water.

Measurement

- 1) Fill the sample in a container
- 2) Wash the sensor module with the water and wipe with tissue
- 3) Put the sensor module in the sample
- 4) Wait the pH is stabilized and read result

Maintenance

- After taking measurements, wash the electrode using pure water; storage it with its cap filled with water.
- When electrode is not to be used for a long period (more than 1 month), store the electrode in dry condition.
- If the tip of the pH electrode is extremely dirty, the speed of its response may slow and it may cause errors in measurement. If the electrode is so dirty that it can be cleaned by rinsing with pure water.

VII. EXPRESION OF REAULTS

- Round the raw value to one decimal place for reporting value.
ex. 10.76 rounded off to one decimal place is 10.8

2 Determination of Conductivity

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- Measured by electric conductivity meter

Type of sample and Range

- This method is applicable to all types of water and waste water samples. The measurement range is defined by the equipment but it is to include 0.1mS/m – 4S/m.

II. PRINCIPLE

- The ability of water to conduct an electric current is known as conductivity or specific conductance and depends on the concentration of ions in solution.
- Conductivity is measured in millisiemens per metre ($1 \text{ mS m}^{-1} = 10 \text{ }\mu\text{S cm}^{-1} = 10 \text{ }\mu\text{mhos cm}^{-1}$).

III. SAMPLING AND SAMPLE PRESERVATION

- The measurement should be made in situ, or in the field immediately after a water sample has been obtained, because conductivity changes with storage time. Conductivity is also temperature-dependent; thus, if the meter used for measuring conductivity is not equipped with automatic temperature correction, the temperature of the sample should be measured and recorded.

IV. APPARATUS

- 1) Beaker 100ml
- 2) Tissue paper
- 3) Conductivity meter (Salinity meter)

V. REAGENTS

- 1) Pure water

VI. PROCEDURE

Calibration

Calibration is normally unnecessary

Measurement

- 1) Fill the sample in a container
- 2) Wash the sensor module with the water and wipe with tissue
- 3) Put the sensor module in the sample
- 4) Wait the conductivity is stabilized and read result

Maintenance

- After taking measurements, wash the electrode using pure water; wipe off the water from the electrode with tissue paper and storage it with its cap on.
- When electrode is not to be used for a long period (more than 1 month), store the electrode after performing the following procedure
- Remove electrode from the Conductivity meter
- Use pure water to wash away any sample solution that may have adhere to the electrode

- Wash inside of the electrode protective cap with pure water then shaking out the water, fill the cap with pure enough pure water to soak the sponge
- Place the electrode protective cap on the electrode.
- If the tip of the conductivity electrode is extremely dirty, the speed of its response may slow and it may cause errors in measurement. If the electrode is so dirty that it can be cleaned by rinsing with pure water.

VII. EXPRESION OF REAULTS

- Conductivity has many units (mS/m, μ S/m, S/m). Relationship among units are, “1S/m = 1000mS/m = 10000 μ S/cm)
- To report the result, “mS/m” or “S/m” is recommendable units in the International System of Units (SI).
- Three (3) digits significant figures are recommended.

3 Determination of Dissolved Oxygen (DO) - Electrochemical probe method

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- ISO 5814:1990 Water quality - Determination of dissolved oxygen - Electrochemical probe method

Type of sample and Range

- This method is applicable to all types of water and waste water samples. The measurement range is 0.2mg/l or more.

II. PRINCIPLE:

- Dissolved oxygen (DO) levels in natural and wastewaters depend on the physical, chemical, and biochemical activities in the water body. The analysis for DO is a key test in water pollution and waste treatment process control.

III. SAMPLING AND SAMPLE PRESERVATION:

- The measurement should be made in situ, or in the field immediately after a water sample has been obtained, because DO changes with storage time.

IV. APPARATUS

- 1) Beaker 100ml
- 2) Tissue paper
- 3) Conductivity meter (Salinity meter)

V. REAGENTS

- 1) Pure water
- 2) Sodium sulfite (Na_2SO_3) solution: Dissolve about 25g of sodium sulfite in water, and add water to make total 500ml. Prepare when it is needed. This solution is used for zero adjustment.

VI. PROCEDURE

Calibration

- Follow manufacturer's calibration procedure exactly to obtain guaranteed precision and accuracy. Generally, calibrate membrane electrodes by reading against air or a sample of known DO concentration (determined by iodometric method) as well as in a sample with zero DO.
- DO values in the saturated pure water are shown in the next page.

Measurement

- Follow all precautions recommended by manufacturer to insure acceptable results.
- Take care in changing membrane to avoid contamination of sensing element and also trapping of minute air bubbles under the membrane, which can lead to lowered response and high residual current.
- Stir the probe in the water sample to provide sufficient sample flow across membrane surface which overcome erratic response.

Maintenance

- After taking measurements, wash the electrode using pure water; wipe off the water from the electrode with tissue paper and storage it with its cap on.

VII. EXPRESION OF REAULTS

- Report the result to one decimal place.
ex. 10.76mg/l rounded off to one decimal place is 10.8mg/l

Appendix

Saturated dissolved oxygen concentration in pure water (mg/L)

Pressure: 1013hPa Oxygen: 20.9% in steam saturation atmosphere

°C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	Salinity correction *
0	14.16	14.12	14.08	14.04	14.00	13.97	13.93	13.89	13.85	13.81	0.00153
1	13.77	13.74	13.70	13.66	13.63	13.59	13.55	13.51	13.48	13.44	0.00148
2	13.40	13.37	13.33	13.30	13.26	13.22	13.19	13.15	13.12	13.08	0.00144
3	13.05	13.01	12.98	12.94	12.91	12.87	12.84	12.81	12.77	12.74	0.00140
4	12.70	12.67	12.64	12.60	12.57	12.54	12.51	12.47	12.44	12.41	0.00135
5	12.37	12.34	12.31	12.28	12.25	12.22	12.18	12.15	12.12	12.09	0.00131
6	12.06	12.03	12.00	11.97	11.94	11.91	11.88	11.85	11.82	11.79	0.00128
7	11.76	11.73	11.70	11.67	11.64	11.61	11.58	11.55	11.52	11.50	0.00124
8	11.47	11.44	11.41	11.38	11.36	11.33	11.30	11.27	11.25	11.22	0.00120
9	11.19	11.16	11.14	11.11	11.08	11.06	11.03	11.00	10.98	10.95	0.00117
10	10.92	10.90	10.87	10.85	10.82	10.80	10.77	10.75	10.72	10.70	0.00113
11	10.67	10.65	10.62	10.60	10.57	10.55	10.53	10.50	10.48	10.45	0.00110
12	10.43	10.40	10.38	10.36	10.34	10.31	10.29	10.27	10.24	10.22	0.00107
13	10.20	10.17	10.15	10.13	10.11	10.09	10.06	10.04	10.02	10.00	0.00104
14	9.98	9.95	9.93	9.91	9.89	9.87	9.85	9.83	9.81	9.78	0.00101
15	9.76	9.74	9.72	9.70	9.68	9.66	9.64	9.62	9.60	9.58	0.00099
16	9.56	9.54	9.52	9.50	9.48	9.46	9.45	9.43	9.41	9.39	0.00096
17	9.37	9.35	9.33	9.31	9.30	9.28	9.26	9.24	9.22	9.20	0.00094
18	9.18	9.17	9.15	9.13	9.12	9.10	9.08	9.06	9.04	9.03	0.00091
19	9.01	8.99	8.98	8.96	8.94	8.93	8.91	8.89	8.88	8.86	0.00089
20	8.84	8.83	8.81	8.79	8.78	8.76	8.75	8.73	8.71	8.70	0.00087
21	8.68	8.67	8.65	8.64	8.62	8.61	8.59	8.58	8.56	8.55	0.00086
22	8.53	8.52	8.52	8.49	8.47	8.41	8.44	8.43	8.41	8.40	0.00084
23	8.38	8.37	8.36	8.34	8.33	8.32	8.30	8.29	8.27	8.26	0.00082
24	8.25	8.23	8.22	8.21	8.19	8.18	8.17	8.15	8.14	8.13	0.00081
25	8.11	8.10	8.09	8.07	8.06	8.05	8.04	8.02	8.01	8.00	0.00079
26	7.99	7.97	7.96	7.95	7.96	7.92	7.91	7.90	7.89	7.88	0.00078
27	7.86	7.85	7.84	7.83	7.82	7.81	7.79	7.78	7.77	7.76	0.00077
28	7.75	7.74	7.72	7.71	7.70	7.69	7.68	7.67	7.66	7.65	0.00076
29	7.64	7.62	7.61	7.60	7.59	7.58	7.57	7.56	7.55	7.54	0.00076
30	7.53	7.52	7.51	7.50	7.48	7.47	7.46	7.45	7.44	7.43	0.00075
31	7.42	7.41	7.40	7.39	7.38	7.37	7.36	7.35	7.34	7.33	0.00075
32	7.32	7.31	7.30	7.29	7.28	7.27	7.26	7.25	7.24	7.23	0.00074
33	7.22	7.21	7.20	7.20	7.19	7.18	7.17	7.16	7.15	7.14	0.00074
34	7.13	7.12	7.11	7.10	7.09	7.08	7.07	7.06	7.05	7.05	0.00074
35	7.04	7.03	7.02	7.01	7.00	6.99	6.98	6.97	6.96	6.95	0.00074
36	6.94	6.94	6.93	6.92	6.91	6.90	6.89	6.88	6.87	6.85	—
37	6.86	6.85	6.84	6.83	6.82	6.81	6.80	6.79	6.78	6.77	—
38	6.76	6.76	6.75	6.74	6.73	6.72	6.71	6.70	6.70	6.69	—
39	6.68	6.67	6.66	6.65	6.64	6.63	6.63	6.62	6.61	6.60	—
40	6.59	6.58	6.57	6.56	6.56	6.55	6.54	6.53	6.52	6.51	—

* DO concentration which should be deducted per chloride ion 100mg/L

G.A. Truesdale et al. "The solubility of Oxygen in Pure Water an Sea-water",
J.Appl. Chem., Vol.5, No.2, P53~62, 1955

4 Determination of Dissolved Oxygen (DO) - Winkler method

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN7324:2004 (ISO5813:1983) Water quality - Determination of dissolved oxygen – Iodometric method

Type of sample and Range

- This method is applicable to all types of water samples. The measurement range is 0.2 mg/l or more.

Interferences

- Nitrites up to a concentration of 15mg/l do not interfere with the determination because they are destroyed by the addition of sodium azide.
- If suspended matter, capable of fixing or consuming iodine, is present, it is preferable to use the electrochemical probe method.

II. PRINCIPLE:

- Dissolved oxygen (DO) levels in natural and wastewaters depend on the physical, chemical, and biochemical activities in the water body. The analysis for DO is a key test in water pollution and waste treatment process control.
- React manganese (II) sulfate with alkaline potassium iodide – sodium azide to produce manganese (II) hydroxide, which is oxidized by dissolved oxygen and changed into manganese (III) hydroxide. Then, add sulfuric acid to dissolve the precipitate, titrate isolated iodine with sodium thiosulfate solution to determine dissolved oxygen.

III. SAMPLING AND SAMPLE PRESERVATION:

- The measurement should be made in situ, or in the field immediately after a water sample has been obtained, because DO changes with storage time.

IV. APPARATUS:

- 1) DO bottle; 100ml
- 2) Burette
- 3) Stirrer
- 4) Magnet
- 5) Pipette (to put 2ml) x2

V. REAGENTS

- 1) Sulphuric acid, solution (1+1):
 - Cautiously add 250ml of concentrated H_2SO_4 to 250ml water, stirring continuously. Concentration is 9mol/l.
- 2) Sulphuric acid, solution, $c(1/2 \text{H}_2\text{SO}_4) = 2 \text{ mol/l}$
- 3) Alkali-iodide-azide reagent:
WARNING – Sodium azide is an extremely strong poison. If nitrites are known to be absent, this reagent may be omitted.
 - Dissolve 35 g NaOH (or 50 g KOH) and 30 g KI (or 27 g NaI) in distilled water and dilute to 50ml.
 - Dissolved 1g sodium azide (NaN_3) in a few milliliters of water.

- Mix the two solutions and dilute to 100 ml.
- Store the solution in a stoppered, brown glass flask.
- After dilution and acidification, this reagent should not show any color in the presence of the indicator solution.
- 4) Manganese (II) sulfate,
 - Dissolve 380 g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ in distilled water, filter, and dilute to 1 L. The MnSO_4 solution should not give a color with starch when added to an acidified potassium iodide (KI) solution.
- 5) Standard potassium iodate solution, $c(1/6 \text{ KIO}_3) = 10 \text{ mmol/l}$
 - Dry a few grams of potassium iodate (KIO_3) at 180°C . Weight $3.567 \pm 0.003 \text{ g}$ and dissolve to 1000 ml.
 - Withdraw 100ml and dilute with water to 1000ml in a volumetric flask.
- 6) Standard sodium thiosulfate: $c(\text{Na}_2\text{S}_2\text{O}_3) = 10 \text{ mmol/l}$
 - Dissolve 2.5 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water.
 - Add 0.4 g solid NaOH and dilute to 1000 ml.
 - Store the solution in a dark glass bottle.
 - **Standardization**—Dissolve approximately 0.5 g KI or NaI, free from iodate, in an erlenmeyer flask with 100 to 150 mL distilled water. Add 2 ml of 2mol/l sulphuric acid solution (2).
 - Mix and add 20.00ml of the standard KIO_3 solution (5). Dilute to about 200 ml and titrate liberated iodine with sodium thiosulfate solution, adding starch toward end of titration, when a pale straw color is reached, and then titrating until complete decoloration.
- The concentration c , expressed in millimole per litre (mmol/l), is given by the equation

$$c = \frac{6 \times 20 \times 1.66}{V}$$
 where V is the volume (ml) of sodium thiosulfate solution used for the titration
- Standardize the solution daily.
- 7) Starch: freshly prepared 10g/l solution

VI. PROCEDURE

Field work

- 1) Fill the DO bottle to overflowing, taking care to avoid any change in the concentration of dissolved oxygen. After elimination of any air bubbles that may be adhering to the glass, immediately fix the dissolved oxygen.
- 2) After sample has been taken, preferably on site, immediately add to the DO bottle, containing the sample, 1ml of the manganese(II) sulfate solution (4) and 2ml of the alkaline reagent (3). Add reagents below the surface using narrow tipped pipettes. Replace the stopper carefully to exclude air bubbles and mix by inverting bottle a few times.
- 3) The DO bottle may be transported to the laboratory.

Measurement

- 1) After the sample arrive at the laboratory, add 1.5ml of the $\text{H}_2\text{SO}_4(1+1)$. Put a magnet in the DO bottle and stir by the stirrer.
- 2) Titrate with the sodium thiosulfate solution (6), adding starch toward end of titration, when a pale straw color is reached, and then titrating until complete decoloration.
- 3) Record the volume of the sodium thiosulfate solution used

VII. EXPRESION OF REAULTS

Calculation

DO content, expressed in milligrams of oxygen per little is given by the formula

$$\text{DO(mg/l)} = \frac{M \times V_2 \times c \times f_1}{4 \times V_1}$$

Where M is the relative molecular mass of oxygen (M=32)

V1 is the volume (ml) of the test sample (=100ml)

V2 is the volume (ml) of sodium thiosulfate solution

C is the actual concentration (m mol/l) of sodium thiosulfate solution

$$f_1 = V_1 / (V_1 - V')$$

V' is the sum of the volumes of reagents (=3ml)

$$f_1 = V_1 / (V_1 - V') = 100 / (100 - 3) = 1.0309$$

$$\begin{aligned} \text{DO(mg/l)} &= \frac{32 \times V_2 \times c \times 1.0309}{4 \times 100} \\ &= 0.082474 \times V_2 \times c \end{aligned}$$

- Report the result to one decimal place.
ex. 10.76mg/l rounded off to one decimal place is 10.8mg/l

5 Determination of Total Suspended Solid (TSS)

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6625:2000 (ISO 11925:1997) Water quality- Determination of suspended solid by filtration through glass-fibre filters

Type of sample and Range

- This method is applicable to all types of water samples. The measurement range is 2 mg/l or more.

Interferences

- Floating oil and other immiscible organic liquids will interfere

II. PRINCIPLE

- A well-mixed, measured portion of a sample is filtered through a standard glass-fiber filter and the residue on filter dried to a constant weight at 103-105 °C. The increase in dish weight represents the total suspended solids.

III. SAMPLING AND SAMPLE PRESERVATION

- Samples shall preferably be taken in bottles of transparent material. Avoid filling the bottles completely, to allow efficient mixing by shaking the bottle.
- Analyze samples for the determination of suspended solids as soon as possible after sampling, preferably within 4h. Store samples which cannot be analyzed within 4h in the dark at below 8°C, but do not allow the sample to freeze. Interpret results obtained for samples that have been stored more than 24 h with caution.
- Samples for the determination of suspended solids shall not be preserved by the addition of any additives.

IV. APPARATUS

- 1) Drying oven
- 2) Filtration apparatus
- 3) Analytic balance (including disk)
- 4) Dessicator provided with a desiccant containing a color for moisture concentration.
- 5) GF/C filter paper (glass-fiber filter)
- 6) Watch glass

V. REAGENTS

- 1) Pure water

VI. PROCEDURE

- 1) Allow samples to attain room temperature
- 2) Put number on the watch glasses
- 3) Set glass-fiber filter in filtration apparatus.
- 4) Vacuum and wash disk with three successive 20 ml pure water. Then, put the filter on a watch glass.
- 5) Dry the filter in oven at 105°C for 1 hour.

- 6) Cool the filter and watch glass in a desiccator and weigh (b (mg)).
- 7) Set the glass-fiber filter in filtration apparatus again.
- 8) Then serve volume 200 - 1000 (ml) of sample. Select the sample volume so that the dry residue on the filter will be in optimum mass range for the determination, which is 5mg to 50mg. However, avoid sample volumes exceeding 1 litre. To be valid, the result shall be based on a dry residue of at least 2mg.
- 9) Vacuum and wash disk with three successive 20 ml of distilled water.
- 10) Put the filter on the same watch glass
- 11) Dry the filter and watch glass in oven at 105°C for at least 1 hour.
- 12) Cool in desiccator and weigh.
- 13) Repeat to dry and weigh to constant weight (a(mg))

VII. EXPRESSION OF RESULTS

Calculation

$$\text{TSS (mg/l)} = (a(\text{mg}) - b(\text{mg})) \times 1000 / V(\text{ml})$$

Where V: volume of sample (ml)

- Report results below 2 mg/l as “below 2 mg/l” and other results in milligrams per litre, to two significant figures.

6 Determination of Total Dissolved Solid (TDS)

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- American Public Health Association, Standard Methods for the Examination of Water and Wastewater 20th Edition (SMEWW) 2540-Solid-C Total Dissolved Solids Dried at 180°C

Type of sample and Range

- This method is applicable to all types of water samples.

Interferences

- Highly mineralized waters with a considerable calcium, magnesium, chloride, and/or sulfate content may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.

II. PRINCIPLE

- A well-mixed, measured portion of a sample is filtered through a standard glass-fiber filter and the filtrate is evaporated to dryness in a weighed dish and dried to a constant weight at 180 °C. The increase in dish weight represents the total dissolved solids.

III. SAMPLING AND SAMPLE PRESERVATION

- Samples shall preferably be taken in bottles of transparent material. Avoid filling the bottles completely, to allow efficient mixing by shaking the bottle.
- Analyze samples for the determination of suspended solids as soon as possible after sampling, preferably within 4h. Store samples which cannot be analyzed within 4h in the dark at below 8°C, but do not allow the sample to freeze. Interpret results obtained for samples that have been stored more than 24 h with caution.
- Samples for the determination of suspended solids shall not be preserved by the addition of any additives.

IV. APPARATUS

- 1) Drying oven, for operation at 180 ± 2 °C.
- 2) Evaporating dish
- 3) Filtration apparatus
- 4) Glass-fiber filter disk
- 5) Analytic balance
- 6) Dessicator provided with silica gel for drying inside
- 7) Evaporating dish

V. REAGENTS

- 1) Pure water

VI. PROCEDURE

- 1) Dry evaporating dish in an oven at 180°C for 1 hour.
- 2) Cool the dish in dessicator and weigh (B mg).
- 3) Set glass-fiber filter disk in filtration apparatus.

- 4) Vacuum and wash disk with three successive 20 ml pure water.
- 5) Take known volume (V ml ; 200ml-1000ml) of sample and place it on filtration apparatus
- 6) Vacuum and transfer filtrate to a weighed clean evaporating dish
- 7) Transfer filtrate to a weighed evaporating dish
- 8) Dry the evaporating dish in an oven at 180°C for at least an hour
- 9) Cool in desiccators and weigh (A mg)
- 10) Repeat to dry and weigh to constant weight

VII. EXPRESION OF REAULTS

Calculation

$$\text{TDS(mg/l)} = (A(\text{mg})-B(\text{mg})) \times 1000/ V(\text{ml})$$

Where V: volume of sample (ml)

- Report results below 2 mg/l as “below 2 mg/l” and other results in milligrams per little, to two significant figures.

7 Determination of COD(Cr)

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- American Public Health Association, Standard Methods for the Examination of Water and Wastewater 20th Edition (SMEWW) 5220-COD-D Closed Reflex, Colorimetric Method

Type of sample and Range

- It is applicable to water with a COD value of between 10 mg/l and 900 mg/l. The chloride content must not exceed 900 mg/l. A water sample which is in accordance with these conditions is used directly for analysis.
- If the COD value exceeds 700 mg/l, the water sample is diluted. For greatest accuracy it is preferable that the COD value of the sample is in the range of 300 mg/l to 600 mg/l.

Interferences

- For this procedure to be applicable, all visible light-absorbing interference must be absent or be compensated for. This includes insoluble suspended matter as well as colored components.

II. PRINCIPLE

- The definition of COD(Cr) is as follows.
The concentration of oxygen equivalent to the amount of dichromate consumed by dissolved and suspended matter when a water sample is treated with that oxidant under defined conditions.
- When a sample is digested, the dichromate ion oxidizes COD material in the sample. This results in the change of chromium from the hexavalent (VI) state to the trivalent (III) state. Both of these chromium species are colored and absorb in the visible region of the spectrum. The dichromate ion ($\text{Cr}_2\text{O}_7^{2-}$) absorbs strongly in the 400-nm region, where the chromic ion (Cr^{3+}) absorption is much less. The chromic ion absorbs strongly in the 600-nm region, where the dichromate has nearly zero absorption. In 9M sulfuric acid solution, the approximate molar extinction coefficients for these chromium species are as follows: Cr^{3+} – 50 L/mole cm at 604 nm; $\text{Cr}_2\text{O}_7^{2-}$ – 380 L/mole cm at 444 nm; Cr^{3+} – 25L/mole cm at 426 nm. The Cr^{3+} ion has a minimum in the region of 400 nm. Thus a working absorption maximum is at 420 nm.
- For COD values between 100 and 900 mg/L, increase in Cr^{3+} in the 600-nm region is determined. Higher values can be obtained by sample dilution. COD values of 90 mg/L or less can be determined by following the decrease in $\text{Cr}_2\text{O}_7^{2-}$ at 420 nm. The corresponding generation of Cr^{3+} gives a small absorption increase at 420 nm, but this is compensated for in the calibration procedure.

III. SAMPLING AND SAMPLE PRESERVATION

- Preferably collect samples in glass bottles. Test unstable samples without delay. If delay before analysis is unavoidable, preserve sample by acidification to $\text{pH} \leq 2$ using conc H_2SO_4 .
- Blend (homogenize) all samples containing suspended solids before analysis. If COD is to be related to BOD, TOC, etc., ensure that all tests receive identical pretreatment. Make preliminary dilutions for wastes containing a high COD to reduce the error inherent in measuring small sample volumes.

IV. APPARATUS:

- 1) Digestion vessels: Preferably use borosilicate culture tubes, 16- × 100-mm, 20- × 150-mm, or 25- × 150-mm, with TFE-lined screw caps. Alternatively, use borosilicate ampoules, 10-mL capacity, 19- to 20-mm diam. Digestion vessels with premixed reagents and other accessories are available from commercial suppliers.
- 2) Block heater or similar device to operate at $150 \pm 2^\circ\text{C}$, with holes to accommodate digestion vessels. Use of culture tubes probably requires the caps to be outside the vessel to protect caps from heat. CAUTION: Do not use an oven because of the possibility of leaking samples generating a corrosive and possibly explosive atmosphere. Also, culture tube caps may not withstand the 150°C temperature in an oven.

V. REAGENTS

- 1) Digestion solution, high range: Add to about 500 ml distilled water 10.216 g $\text{K}_2\text{Cr}_2\text{O}_7$ primary standard grade, previously dried at 150°C for 2 h, 167 ml conc H_2SO_4 , and 33.3 g HgSO_4 . Dissolve, cool to room temperature, and dilute to 1000 ml.
- 2) Digestion solution, low range: Prepare as in 1), but use only 1.022 g potassium dichromate.
- 3) Sulfuric acid reagent: Add Ag_2SO_4 , reagent or technical grade, crystals or powder, to conc H_2SO_4 at the rate of 5.5 g $\text{Ag}_2\text{SO}_4/\text{kg}$ H_2SO_4 . Let stand 1 to 2 d to dissolve. Mix.
- 4) Potassium hydrogen phthalate (KHP) standard: $\text{HOOC}_6\text{H}_4\text{COOK}$: Lightly crush and then dry KHP to constant weight at 110°C . Dissolve 425 mg in distilled water and dilute to 1000mL. KHP has a theoretical COD of 1.176 mg O_2/mg and this solution has a theoretical COD of 500 μg O_2/ml . This solution is stable when refrigerated, but not indefinitely. Be alert to development of visible biological growth. If practical, prepare and transfer solution under sterile conditions. Weekly preparation usually is satisfactory.

VI. PROCEDURE

- 1) Wash digestion vessels and caps with 20% H_2SO_4 before first use to prevent contamination.

WARNING

Wear face shield and protect hands from heat produced when contents of vessels are mixed. Mix thoroughly before applying heat to prevent local heating of vessel bottom and possible explosive reaction.

- 2) Blend (homogenize) all samples containing suspended solids before analysis.
- 3) (In case of 16- × 100-mm) Place 2.5ml of sample, 1.5 ml of Digestion solution in the digestion vessels. Carefully run 3.5ml of Sulfuric acid reagent down inside of tube so an acid layer is formed under the digestion solution layer. Make volumetric measurements as accurate as practical; The most critical volumes are of the sample and digestion solution. The use of micro-pipettes with non-wetting (polyethylene) pipet tips is practical and adequate.
- 4) At least one blank sample is to be prepared and more than one standard in the tube. It is critical that the volume of each component be known and that the total volume be the same for each reaction tube.
- 5) Tightly cap tubes, and invert each several times to mix completely.
- 6) Place tubes in block digester preheated to 150°C and reflux for 2 h behind a protective shield.

WARNING

These sealed tubes may be under pressure from gases generated during digestion. Wear face and hand protection when handling. If sulfuric acid is omitted or reduced in concentration, very high and dangerous pressures will be generated at 150°C .

- 7) Cool to room temperature and place tubes in test tube rack. Some mercuric sulfate may precipitate out but this will not affect the analysis.

Measurement of dichromate reduction:

- 1) Cool sample to room temperature slowly to avoid precipitate formation. Once samples are cooled, vent, if necessary, to relieve any pressure generated during digestion.
 - 2) Mix contents of reaction vessels to combine condensed water and dislodge insoluble matter. Let suspended matter settle and ensure that optical path is clear.
 - 3) Measure absorption of each sample blank and standard at selected wavelength (420 nm or 600nm). For COD values between 90 and 830 mg/L, increase in Cr^{3+} in the 600-nm region is determined. Higher values can be obtained by sample dilution. COD values of 150 mg/L or less can be determined by following the decrease in $\text{K}_2\text{Cr}_2\text{O}_7^-$ at 420 nm.
- High Range, measuring at 600 nm, (measuring absorbance of Chromate)
Use an undigested blank as reference solution. Analyze a digested blank to confirm good analytical reagents and to determine the blank COD; subtract blank COD from sample COD. Alternately, use digested blank as the reference solution once it is established that the blank has a low COD.
 - Low Range, measuring at 420 nm, (measuring absorbance of Dichromate);
Use distilled water as a reference solution. Measure all samples, blanks, and standards against this solution. The absorption measurement of an undigested blank containing dichromate, with distilled water replacing sample, will give initial dichromate absorption. Any digested sample, blank, or standard that has a COD value will give lower absorbance because of the decrease in dichromate ion. Analyze a digested blank with distilled water replacing sample to ensure reagent quality and to determine the reagents' contribution to the decrease in absorbance during a given digestion. The difference between absorbances of a given digested sample and the digested blank is a measure of the sample COD. When standards are run, plot differences of digested blank absorbance and digested standard absorbance versus COD values for each standard

Preparation of calibration curve

- Prepare at least five standards from potassium hydrogen phthalate solution with COD equivalents to cover each concentration range. Make up to volume with distilled water; use same reagent volumes, tube size, and digestion procedure as for samples. Prepare calibration curve for each new lot of tubes or when standards prepared in differ by $\geq 5\%$ from calibration curve. Curves should be linear. However, some nonlinearity may occur, depending on instrument used and overall accuracy needed.

VII. EXPRESION OF REAULTS

- Report results below 10 mg/l as "below 10 mg/l" and other results in milligrams per little, to two significant figures.

8 Determination of BOD

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6001:1995 (ISO 5815-1:2003) Water quality - Determination of biochemical oxygen demand after n days (BOD n)

Type of sample and Range

- This method is applicable to all types of water samples. The measurement range is 0.5 mg/l or more.

Interferences

- The test can be influenced by the presence of various substances. Those which are toxic to microorganisms, for example bactericides, toxic metals or free chlorine, will inhibit biochemical oxidation. The presence of algae or nitrifying microorganisms can produce artificially high results.

II. PRINCIPLE

- The sample of water to be analyzed is pretreated and diluted with varying amounts of a dilution water rich in dissolved oxygen and containing a seed of aerobic microorganisms, with suppression of nitrification.
- The sample is incubated at 20 °C for a defined period, 5 days, in the dark, in a completely filled and stoppered bottle. The dissolved oxygen concentration is determined before and after incubation, and the mass of oxygen consumed per liter of sample is calculated.

III. SAMPLING AND SAMPLE PRESERVATION

- Begin analysis within 6h of collection, when this is not possible because the sampling site is distant from the laboratory.
- Store at or below 4°C and report length and temperature of storage with the results. BOD analysis should be started within 24 hours after taking samples.

IV. APPARATUS:

- 1) Incubation Bottles (BOD bottle): Use glass bottles having 100 ml. clean bottles with a detergent, rinse thoroughly and drain before use. This bottles having a ground glass-stopper and a flared mouth.
- 2) Incubator: Thermostatically controlled at 20 ± 1 °C
- 3) DO meter: Dissolved oxygen probe calibrating by using saturated oxygen water. (Saturated water prepared by hand Shaking technique). Otherwise, apparatus for Winkler method are to be prepared.

V. REAGENTS

Prepare reagents in advance but discard if there is any sign of precipitation or biological growth in the stock bottles.

- 1) Phosphate Buffer Solution: Dissolve 0.85 g KH_2PO_4 , 2.175 g K_2HPO_4 , 4.466 g Na_2HPO_4 and 0.17gm NH_4Cl in about 50 ml distilled water and dilute to 100 ml volumetric flask.
- 2) Magnesium Sulfate Solution: Dissolve 2.25 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and up to mark in 100 ml volumetric flask.
- 3) Calcium chloride Solution: Dissolve 2.75 gm CaCl_2 in 100 ml volumetric flask and up to mark with distilled water.

- 4) **Iron(III) Chloride Solution:** Dissolve 0.025g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 100 ml volumetric flask and up to mark with distilled water.

VI. PROCEDURE

Check pH of all samples before testing. pH of sample should be around 7 (6.5 - 7.5) for BOD analysis. Neutralize, if necessary, by 1 N- H_2SO_4 or 1 N-NaOH.

Preparation

- 1) **Preparation of Saturated Water:** Saturate distilled water with DO by shaking in a partially filled bottle/separated funnel. An aeration pump is more suitable for preparation of saturated Oxygen water. The duration of aeration is required around 2 ~ 3 hours.
- 2) **Preparation of dilution water:** Saturated oxygen water in a Suitable bottle/ beaker and add 1 ml each of phosphate buffer, MgSO_4 , CaCl_2 and FeCl_3 solutions per 1000ml of water.
- 3) **DO probe calibrating:** DO probe calibrating by saturated water (at 20°C saturated water should DO 8.84 mg/L) and compare with reading and temperature (See the chart).
- 4) **Preparation of Sample:** After control pH, sample is needed to be diluted by dilution water. Dilution water contains nutrients such as nitrogen, phosphorus and trace metals for bacterial growth.

Seeding Method

- BOD analysis is the method of measuring organic compounds using bacteria. But basically drinking water (Deep well water) contains no bacteria. In that case the seeding method (adding bacteria to dilution water of BOD) is required for the analysis.

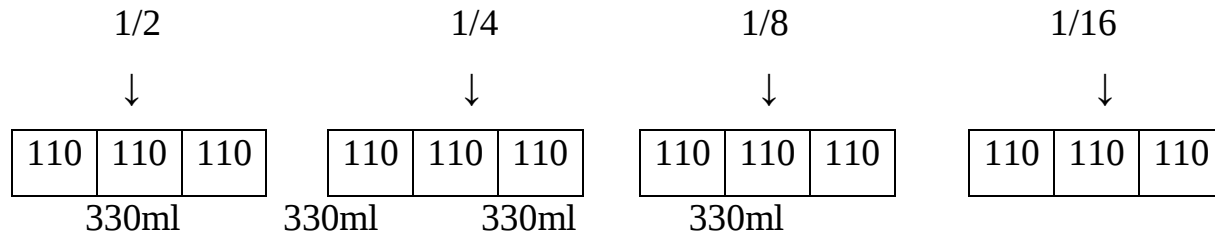
Seed source/ control

- It is necessary to have a population of micro organism capable of oxidizing the biodegradable organic matter in the sample. Determine the BOD of the seeding material as for any other sample. This is the seed control. From the value of the seed, control and knowledge of the seeding material dilution determine seed DO up take. BOD of diluted sample contain should be more than 0.6 mg/L seed source. As example if BOD of seeding source is 100 mg/L, more than 6ml of it should be taken in 1 L diluted water.

Sample dilution technique

- Make several dilutions of sample that will result in a residual DO of at least 1 mg/L and a DO uptake of at least 2 mg/L after 5 day's incubation. Five dilutions are recommended unless experiences with a particular sample shows that use of a smaller number of dilutions procedure at least two bottles giving acceptable minimum DO deletion and residual limits.
- Prepare dilution either in graduated cylinders or volumetric glassware and then transfer to BOD bottles or pressure directly in BOD bottles. Either diluted method can be combined with any DO measurement technique. The number of bottles to be prepared for each dilution depends on the DO technique and the number of replicates desired.
- When using graduated cylinders or volumetric flasks to prepare dilution and when seeding is necessary add seed either directly to dilution water or individual cylinders or flasks before dilution.
- **Why series Dilution is needed?**
- Concentration of BOD depends on different source of water. We do not know which type of samples/water contains how much BOD. Another concern it needed more than five days with continuous electricity supply to control temperature at $20^\circ\text{C} \pm 1$ for BOD analysis. To avoid re-analysis and get acceptable result we need series dilution of sample. So that we can compare between different types of dilution of same sample and get acceptable result.

Dilution technique



- First we consider how much volume of sample is need for first dilution factor. In this case first dilution factor is $\frac{1}{2}$. For this, sample need $330 \times \frac{1}{2} = 165$ ml. Then for whole series sample will need $\frac{1}{2}$ of the previous.
i.e.

For $\frac{1}{4}$, sample need $165/2 = 82.5$ ml

For $\frac{1}{8}$, sample need $82.5/2 = 41.25$ ml

For $\frac{1}{16}$, sample need $41.25/2 = 20.625$ ml

Then add all volume of sample which will be needed.

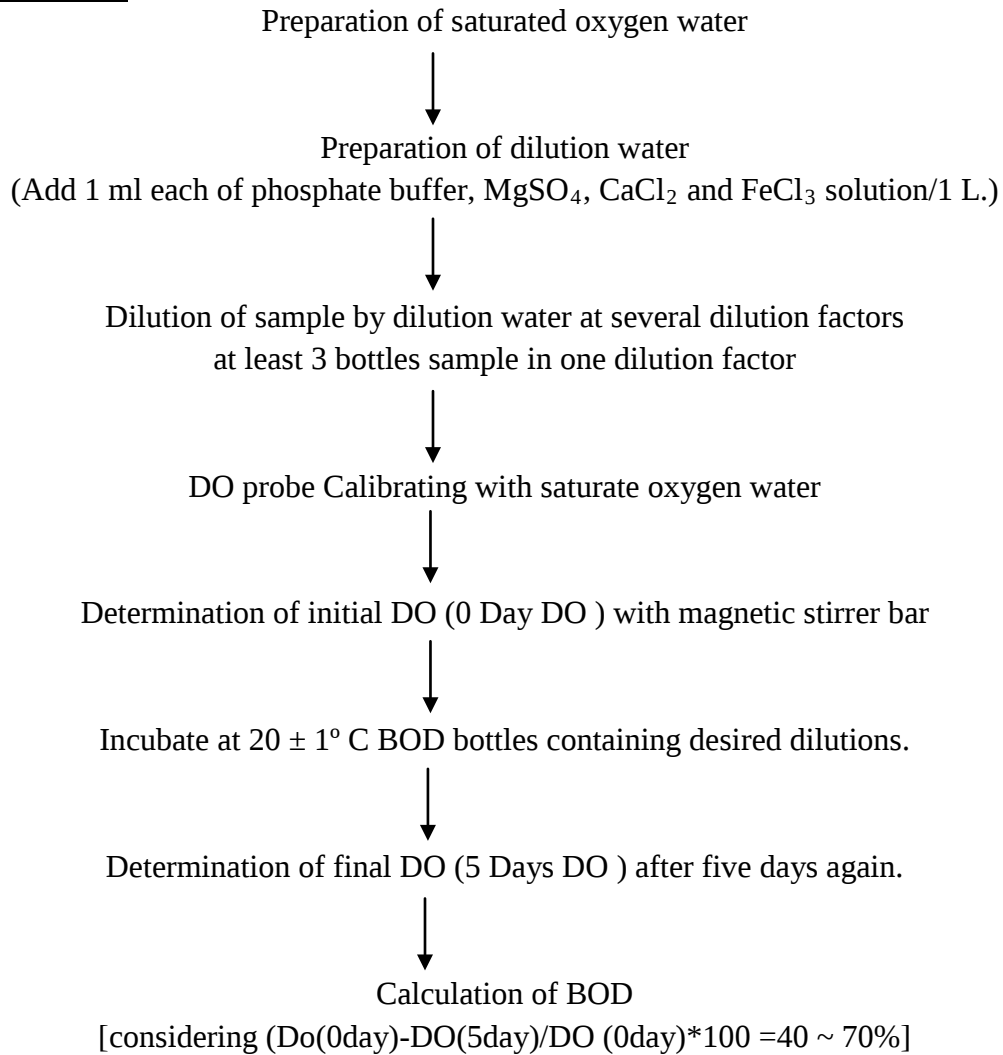
$(165 + 82.5 + 41.25 + 20.625) = 309.375$ ml

Then multiply first dilution factor, in this case first volume will be $309.375 \text{ ml} \times 2 = 618.75$ ml

Determination of initial DO

- If the sample contains materials that react rapidly with DO, determine initial DO immediately. After filling BOD bottle with diluted sample. Place a clean magnetic stirring bar in each bottle to mix contents before making DO measurement. DO not remove the magnets until the test is complete.
- Incubate at 20 ± 1 °C, BOD bottles containing desired dilutions. Five days has been accepted as the standard incubation period.
- Determination of final DO; After five days incubation, determine DO in sample again with magnetic stirring bar in each bottle.

Flow chart of analysis



VII. EXPRESION OF REAULTS

Calculation

$$BOD_5 (mg / l) = \frac{D_1 - D_2}{P}$$

Where,

D₁ = DO of diluted sample immediately after preparation mg/L.

D₂ = DO of diluted sample after 5 days incubation at 20 °C , mg/L.

P = Fraction of sample used.

When dilution water is seeded:

$$BOD_5 (mg / l) = \frac{(A_1 - A_2) - (B_1 - B_2) \times f}{P}$$

Where
 A1 = DO of diluted by seeded diluted water
 A2 = DO of diluted by seeded diluted water after 5 days incubation
 B1= DO of seed control before incubation, mg/L
 B2 = DO of seed control after incubation, mg/L
 f = Ratio of seed in diluted sample to seed in seed control
 = (% seed in diluted sample) / (% seed in seed control)
 P = Fraction of sample used.

- Report the result to one decimal place.
 ex. 10.76mg/l rounded off to one decimal place is 10.8mg/l

QA/QC (Quality Control and Quality Assurance)

Acceptable range of DO consumption

To secure good quality data, it is required to follow Japanese standard Method.

- Japanese Standard Method
 - Consumption rate should be between 40 and 70 % of initial DO(DO(0), a)
- Standard Methods for the Examination of Water and Wastewater (SMWW)
 - Residual DO (DO(5), b) at least 1.0 mg/L
 - DO depletion (DO(0) – DO(5), a-b) at least 2.0 mg/L

[Example]

(mg/L)	1	2	3	4
DO(0), a	8.84	8.84	8.84	8.84
DO(5), b	6.84	5.30	2.65	1.00
a – b	2.00	3.54	6.19	7.84
(a – b) / a x100 (%)	23	40	70	89
Japanese Standard Method	×	○	○	×
Standard Deviation	1.87			
SMWW	○	○	○	○
Standard Deviation	2.62			

Working range and detection limit

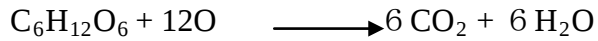
- The working range is equal to the difference between the maximum initial DO (7 to 9 mg/L) and minimum DO residual of 1 mg/L multiplied by the dilution factor. A lower detection limit of 2.0 mg/L is established by the requirement for minimum DO depletion of 2.0 mg/L.
- Though the standard value of BOD in Bangladesh is set 0.2 mg/L, it is impossible to measure such a low value.

Precision and Bias

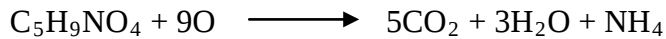
- There is no measurement for establishing bias of the BOD procedure. The Glucose - Glutamic acid (Glutamate) check is intended to be a reference point for evaluation of dilution water quality, seed effectiveness, and analytical technique.
- BOD Standard Solution

Glucose (C₆H₁₂O₆) 150 mg + Glutamate (C₅H₉NO₄) 150 mg in 1 L distilled water

Glucose(C₆H₁₂O₆) (M=180.16) 150 mg = 0.8325 mmol



Glutamate(C₅H₉NO₄) (M=147.13) 150 mg = 1.020 mmol



When you measure BOD of BOD Standard Solution, the result is expected to be **220±10 mg/L** (= around 70 % is decomposed).

If the result was not 220±10 mg/L, it means there are some problem in Dilution Water, Seed Source or your skill.

9 Determination of Ammonia (NH₄)

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 5988: 1995 (ISO 5664 – 1984) – Water quality - Determination of ammonium - Distillation and titration method

Type of sample and Range

- The method is applicable to the analysis of potable water, and most raw and waste waters.
- An ammonium nitrogen content of up to 10 mg in the test portion may be determined. Using a 10 ml test portion, this corresponds to a sample concentration of up to NH₄-N = 1 000 mg/l.
- Limit of detection, using a 250ml test portion is 0.2mg/l

Interferences

- The main possible interference arises from urea which, under the conditions specified, will distil as ammonia thus causing high results. Interference will also arise from volatile amines which will distil and react with the acid during the titration, thus causing high results. Chloramines present in chlorinated water samples will be determined in this manner.

II. PRINCIPLE

- Adjustment of the pH of a test portion to within the range 6.0 to 7.4. Addition of magnesium oxide to produce mildly alkaline conditions, distillation of the liberated ammonia and collection in a receiving flask containing boric acid solution. Titration of the ammonium in the distillate with standard volumetric acid solution using boric acid/indicator solution.

III. SAMPLING AND SAMPLE PRESERVATION

- Laboratory samples shall be collected in polyethylene or glass bottles. They should be analyzed as quickly as possible, or else stored at between 2 and 5 °C until analyzed. Acidification with sulfuric acid to pH <2 may also be used as an aid to preservation, provided that possible contamination of the acidified
- Sample by absorption of any atmospheric ammonia is avoided.

IV. APPARATUS:

- 1) Spectrometer, capable of operating at a wavelength of 655 nm with cells of Optical path length between 10 and 50 mm.
- 2) Distillation apparatus

V. REAGENTS

- 1) **Water** ammonium-free: prepared by one of the following methods.
Add 0.10 + 0.01 ml of sulfuric acid (Q = 1,84 g/ml) to 1 000 + 10 ml of distilled water and redistill in an all glass apparatus. Discard the first 50 ml of distillate, and then collect the distillate in a glass bottle provided with a well-fitting glass stopper. Add about 10 g of strongly acidic cation exchange resin (in the hydrogen form) to each liter of collected distillate.
- 2) **Hydrochloric acid**
- 3) **Hydrochloric acid, standard volumetric solution, c(HCl) = 0.10mol/l**

Prepare this solution by diluting the hydrochloric acid (2). Standardize it by normal analytical procedures. Alternatively, commercial solutions of guaranteed concentration may be used.

- 4) **Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0.02 \text{ mol/l}$**
- 5) **Boric acid/ Indicator, solution**
 - Dissolve $0.5 \pm 0.1 \text{ g}$ of water-soluble methyl red in about 800ml of water and dilute to 1 litre.
 - Dissolve $1.5 \pm 0.1 \text{ g}$ of methylene blue in about 800ml of water and dilute to 1 litre.
 - Dissolve $20 \pm 1 \text{ g}$ of boric acid (H_3BO_3) in warm water. Cool to room temperature. Add $10 \pm 0.5 \text{ ml}$ of the methyl red indicator solution and $2.0 \pm 0.1 \text{ ml}$ of the methylene blue solution and dilute to 1 litre.
- 6) **Bromothymol blue, 0.5 g/l Indicator, solution**
 - Dissolve $0.5 \pm 0.02 \text{ g}$ of bromothymol blue in water and dilute to 1 litre.
- 7) **Hydrochloric acid, $1\%(\text{V/V})$ solution**
 - Dilute $10 \pm 1 \text{ ml}$ of hydrochloric acid (2) to 1 litre with water.
- 8) **Sodium hydroxide solution, 1 mol/l**
 - Dissolve $40 \pm 2 \text{ g}$ of sodium hydroxide in about 500ml of water. Cool to room temperature and dilute to 1 litre.
- 9) **Light magnesium oxide, carbonate-free**
 - Heat the magnesium oxide at 500°C to remove carbonate. If there is no oven to heat at the temperature, just skip the process.
- 10) **Anti-bumping granules**

VI. PROCEDURE

Test portion

- If the approximate ammonium content of the sample is known, select the volume of the test portion according to the following table.

Ammonium concentration (mg/l)	Volume of the test portion (ml)
Up to 10	250
10 to 20	100
20 to 50	50
50 to 100	25

Determination

- 1) Transfer $50 \pm 5 \text{ ml}$ of the boric acid/indicator solution(5) into the receiving flask of the distillation apparatus. Ensure that the delivery tip of the condenser is below the surface of the boric acid solution. Measure the selected volume of test portion (see *Test portion*) into the distillation flask. Formation of the absorbing compound
 - Add a few drops of the bromothymol blue indicator solution (6) and, if necessary, adjust the pH to within the range 6.0 (indicator yellow) to 7.4 (indicator blue) using the sodium hydroxide solution (8) or the hydrochloric acid (7), as appropriate. Then make up the total volume in distillation flask to about 350 ml with the ammonium-free water (1).
 - Add to the distillation flask $0.25 \pm 0.05 \text{ g}$ of the light magnesium oxide (9) and a few anti-bumping granules (10).
 - Immediately attach the distillation flask to the apparatus.

- 2) Heat the distillation flask so that distillate collects at a rate of about 10 ml/min. Stop the distillation when about 200 ml have been collected.
- 3) Titrate the distillate to the purple end-point using the standard volumetric hydrochloric acid (4) and record the volume used.

Blank test

- Carry out a blank test by proceeding as described, but replacing the test portion with about 250 ml of the ammonium-free water (1).

VII. EXPRESION OF REAULTS

Calculation

$$NH_4 - N(mgN / l) = \frac{V_1 - V_2}{V_0} \times c \times 14.01 \times 1000$$

Where,

V_0 = the volume (ml) of test portion (sample)

V_1 = the volume (ml) of the standard volumetric HCl used for titration

V_2 = the volume (ml) of the standard volumetric HCl used for titration in blank test

c = the exact concentration (mol/l) of the standard HCl.

14.01 is the atomic mass, in gram per mole, of nitrogen.

Relationship between Ammonia as nitrogen (NH_4-N), Ammonia (NH_3), and Ammonia ion (NH_4^+) is as follows.

Table. Conversion table

	NH_4-N (mg/l)	NH_3 (mg/l)	NH_4^+ (mg/l)
Ammonia as nitrogen (NH_4-N) =1mg/l	1	1.216	1.288
Ammonia (NH_3) =1mg/l	0.823	1	1.059
Ammonia ion (NH_4^+)=1mg/l	0.777	0.944	1

- Report the result to two decimal place, and two digit significant figures.
ex. 0.0767mg/l rounded off to two decimal place is 0.08mg/l.

10 Determination of Fluoride

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6195-1996(ISO10359-1-1992), Water quality -- Determination of fluoride -- Part 1: Electrochemical probe method for potable and lightly polluted water

Type of sample and Range

- The method is for the determination of dissolved fluoride in fresh, portable and low contaminated water, and some surface waters.
- The method is directly suitable for measuring fluoride concentrations from 0.2 mg/l to 2.0 g/l.
- After addition of a known fluoride, concentration as low as 0.02 mg/l can be detected.
- The method is not suitable for waste waters and industrial effluents.

Interferences

- The electrode will respond directly to hydroxide ions. The formation of HF under acidic conditions will reduce the measured fluoride concentration. Therefore, buffer, all test aliquots to a pH between 5 to 7 to prevent such interference.
- Cations such as calcium, magnesium, iron and aluminum form complexes with fluoride or precipitates to which electrode does not respond. Therefore the buffer solution also contains trans-1,2 diaminoxiclohexan-N,N,N', N'-tetraacetic(CDTA) as a decomplexing agent to free bound fluoride.

II. PRINCIPLE

- When a fluoride ion-selective electrode comes into contact with an aqueous solution containing fluoride ions, a potential difference develops between the measuring electrode and the reference electrode. The value of this potential difference is proportional to the logarithm of the value of the fluoride ion activity in accordance with the Nernst equation.
- Values of pH between 5 and 7 have proved favorable for measurement.
- Fluoride ion-selective electrodes operate between 0.2 mg/l and 2,000 mg/l, and show a linear relationship between the potential and the logarithm of the numerical value of the fluoride activity.

III. SAMPLING AND SAMPLE PRESERVATION

- Samples shall be taken in polyethylene bottles which have been washed thoroughly and rinsed with fluoride-free water. No preservative is normally necessary but the analysis should be performed as soon as possible, preferably within 3 days.

IV. APPARATUS:

- Meter, a millivolt meter with an impedance of not less than $10^{10}\Omega$, capable of resolving potential differences of 0.1 mV or better.
- Fluoride ion-selective electrode shall give stable readings.
- Reference electrode, either a calomel electrode, filled with saturated potassium chloride (KCl) solution.
- Magnetic stirrer.
- Membrane filtration device, with membrane filters of pore size 0.45 μm .
- Polyethylene beaker 100ml.
- Polyethylene measuring cells, of capacity 100ml.

V. REAGENTS

Only use reagents of recognized analytical grade and only distilled water or water of equivalent purity.

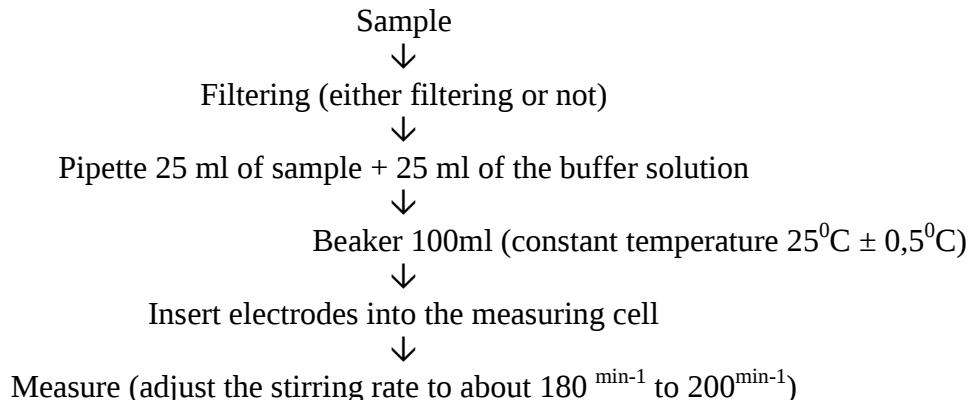
- 4) Sodium hydroxide, $c(\text{NaOH}) = 5\text{ mol/l}$
 - Dissolve $100\text{ g} \pm 0.5\text{ g}$ of sodium hydroxide in water, cool and dilute to 500 ml.
- 5) Total ionic strength adjustment buffer (TISAB)
 - Add 58 g of sodium chloride (NaCl) and 57 ml of glacial acetic acid [$p(\text{CH}_3\text{COOH}) = 1.05\text{ g/ml}$] to 500 ml of water in a 1 liter beaker. Stir until dissolved. Add 150 ml of the sodium hydroxide solution 5mol/l (1) and 4 g of CDTA (axit trans -1,2-diaminocyclohexan-N, N, N',N'-tetraacetic). Continue stirring until all the solids have dissolved and adjust the solution to pH 5.2 with sodium hydroxide solution using a pH meter. Transfer to a 1,000 ml volumetric flask, make up to the mark with water and mix.
- 6) Fluoride, stock solution, 1,000 mg/l
 - Dry a portion of sodium fluoride (NaF) at 150°C for 4h and cool in a desiccator.
 - Dissolve $2.210\text{ g} \pm 0.001\text{ g}$ of the dried material in water contained in a 1000 ml volumetric flask. Make up to the mark with water and mix.
- 3)-1 Fluoride, working standard solution I, 10mg/l
 - Pipette 1ml of fluoride stock solution (3) into a 100 ml volumetric flask, make up to the mark with water and mix.
- 3)-2 Fluoride, working standard solution II, 5mg/l
 - Pipette 0.5ml of fluoride stock solution (3) into a 100 ml volumetric flask, make up to the mark with water and mix
- 3)-3 Fluoride, working standard solution III, 1mg/l
 - Pipette 10 ml of fluoride stock solution (3.1) into a 100 ml volumetric flask, make up to the mark with water and mix
- 3)-4 Fluoride, working standard solution IV, 0.5mg/l
 - Pipette 10 ml of fluoride stock solution II (3.2) into a 100 ml volumetric flask, make up to the mark with water and mix
- 3)-5 Fluoride, working standard solution V, 0.2mg/l
 - Pipette 2 ml of fluoride stock solution I (3.1) into a 100 ml volumetric flask, make up to the mark with water and mix

VI. PROCEDURE

Preparation for measurement

- To accelerate the establishment of the equilibrium potential, condition the electrode prior to measurement in the following way: immerse the electrode for 1h in the cell containing the fluoride with concentration 0.2 mg/l .Then the electrode is ready for use.

Flow chart of measurement



Calibration

- Preparation of reference solutions as indicated in the following Table.

Order of volumetric flash	1	2	3	4
V fluoride 1000 mg/l (ml)	-	-	-	10
V fluoride 100 mg/l (ml)	-	-	10	-
V fluoride 10 mg/l (ml)	2	10	-	-
V volumetric flash (ml)	100	100	100	100
C _a (mg/l)	0,2	1	10	100

VII. EXPRESION OF REAULTS

- The formula for calculating of concentration of F⁻ is as follows,
Nernst equation

$$E = E_0 + \left[\frac{2.303RT}{zF} \right] \times \log a$$

Where

E: cell potential (electromotive force)

E₀: standard cell potential at the temperature of interest

R: Gas Constant

T: absolute temperature

F: Faraday Constant

Z: number of moles of electrons transferred in the cell reaction or half-reaction

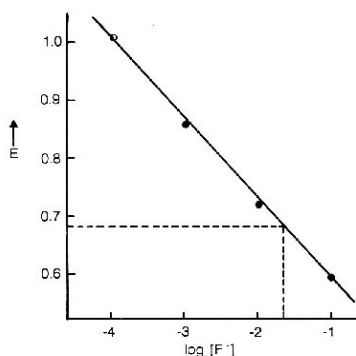
a: activity of ion

$$a = f \cdot c$$

f: activity coefficient

c: concentration of ion.

- The Calibration curve goes like the following Fig. derived from Nernst Equation.



- After the measurement, plot the calibration values on semi-logarithmic paper, with the fluoride concentrations, in milligrams per litre, on the abscissa and the cell potential, in millivolts, on the ordinate and establish the regression line.
- Read the value for the samples by using the regression line and express the mass concentration of fluoride in milligram per litre.

11 Determination of Nitrite Nitrogen

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6178 : 1996 (ISO 6777:1984) Water quality - Determination of nitrite - Molecular absorption spectrometric method

Type of sample and Range

- This International Standard specifies a molecular absorption spectrometric method for the determination of nitrite in potable, raw and waste water.
- A nitrite nitrogen concentration of up to 0,25 mg/l can be determined when using the maximum volume (40 ml) of test portion.
- When using cells of optical path length 40 mm and a test portion of 40 ml, the limit of detection has been determined to lie within the range $N = 0,001$ to $0,002$ mg/l.

II. PRINCIPLE

- Reaction of nitrite in the test portion with 4-aminobenzene sulfonamide reagent in the presence of orthophosphoric acid at pH 1,9 to form a diazonium salt which forms a pink-coloured dye with N-(1-naphthyl)-1,2-diaminoethane dihydrochloride (added with the 4-aminobenzene sulfonamide reagent).
- Measurement of the absorbance at 540nm.

III. SAMPLING AND SAMPLE PRESERVATION

- Laboratory samples should be collected in glass bottles and should be analyzed as soon as possible within 24 h of collection.
- Storage of the samples at 2 to 5 °C may preserve many types of sample, but this should be verified.

IV. APPARATUS:

- All glassware shall be carefully cleaned using approximately 2 mol/l hydrochloric acid and then rinsed thoroughly with water.
- 1) Ordinary laboratory glassware
 - 2) Spectrometer, suitable for measurements at a wavelength of 540nm, together with cells of optical path length between 10 and 50 mm.

V. REAGENTS

- 1) **Orthophosphoric acid (15 mol/l) (H_3PO_4)**
- 2) **Orthophosphoric acid (1.5 mol/l)**
 - Take 25 ml of Orthophosphoric acid (15mol/l) with pipet into 150 ml of water and cool to room temperature. Transfer this solution to 250 ml of volumetric flask and dilute to the marked line with water.
 - Store this solution in dark bottle.
 - This solution is stable for at least 6 months.
- 3) **Color forming reagent**
 - Take 100 ml of orthophosphoric acid (1.5mol/l) into 500 ml of water. Dissolve 40 ± 0.5 g of 4-aminobenzenesulfonamide ($C_6H_8N_2O_2S$) into this solution.

- Dissolve 2 ± 0.02 g of N-(1-Naphthyl)-1,2- diaminoethane dihydrochloride ($C_{12}H_{16}Cl_2N_2$) into this solution. Transfer this solution into 1000 ml of volumetric flask and dilute to marked line with water.
- Store this solution in an amber bottle.
- This solution is stable for one month in 2 to 5 °C condition.
- 4) **Nitrite standard solution (100 mg/l)**
 - Dry sodium nitrite at 105 °C for 2 hours. Take 0.4922 ± 0.0002 of this salt into about 750 ml of water and dissolve. Transfer quantitatively to a 1000ml one-mark volumetric flask. Dilute to the marked line with water.
 - Store this solution in dark bottle and keep in 2 to 5 °C.
 - This solution is stable for one month.
- 5) **Nitrite standard solution (1 mg/l)**
 - Transfer, by means of a pipette, 10 ml of the standard nitrite solution (100mg/l) to a 1000 ml one-mark volumetric flask and dilute to the mark with water.
 - Prepare this solution each day as required, and discard after use.

VI. PROCEDURE

Calibration

- Take 0, 1, 2, 5 and 10 ml of nitrite standard solution (1 mg/L) into 50 ml of volumetric flask respectively. Dilute them into about 40 ml with water. Proceed with measurement procedure.
- Nitrite amount of these solutions is 0, 1, 2, 5 and 10µg nitrite respectively.
-

Preparation of reference solutions:

Measurement

sample maximum 40 mL (*1) ---> 50 mL volumetric flask

|

dilute <--- water ---> 40 mL

|

| <--- 1 mL color forming reagent

mix

|

dilute <--- water ---> marked line

|

leave 20 min.

|

measurement spectrophotometer

at 540 nm

reference:

(*1) if sample contains suspended solids, wait for settling or filter with glass fiber paper

Blank test

Instead of sample solution, 40 mL of water is used in measurement procedure.

VII. EXPRESION OF REAULTS

Calculation

$$\text{NO}_2 \text{ (mg/L)} = M - M(b) / V$$

where, M: amount of nitrite in sample solution (µg)

M(b): amount of nitrite in blank (µg)

V: sample volume (mL)

Table. Conversion table

	µg N/mL	µg NO ₂ /mL
1 µg N/mL	1	3.29
1 µg NO ₂ /mL	0.304	1

VIII. OTHERS

Safety information

- Color forming reagent is harmful.
- Avoid contacting this solution through skin.

12 Determination of Nitrate Nitrogen

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6180:1996 (ISO 7890-3:1998) Water quality - Determination of nitrate - Part 3 Spectrometric method using sulfosalicylic acid

Type of sample and Range

- The method is suitable for application to raw and potable water samples.
- Up to a nitrate nitrogen concentration, N of 0,2 mg/l using the maximum test portion volume of 25 ml. The range can be extended upwards by taking smaller test portions.
- Using cells of optical path length 40 mm and a 25 ml test portion volume the limit of detection lies within the range $N = 0,003$ to $0,013$ mg/l.

Interferences

Major interference in this test is as follows.

- Chloride (Cl^-) ion, ortho phosphate (PO_4^{3-}) ion, Mg, Mn and hydrogen carbonate (HCO_3^-) ion

II. PRINCIPLE:

- Spectrometric measurement of the yellow compound formed by reaction of sulfosalicylic acid (formed by addition to the sample of sodium salicylate and sulfuric acid) with nitrate and subsequent treatment with alkali.
- Disodium dihydrogen ethylenedinitrilotetraacetate (EDTA Na_2) is added with the alkali to prevent precipitation of calcium and magnesium salts. Sodium azide is added to overcome interference from nitrite.

III. SAMPLING AND SAMPLE PRESERVATION:

- Laboratory samples should be collected in glass bottles and should be analyzed as soon as possible after collection. Storage of samples at between 2°C and 5°C may preserve many types of sample, but checks should be made to confirm this with each sample type.

IV. APPARATUS:

- 1) Water bath
- 2) Evaporation dish (50 mL)
When the dishes are new, or not in regular use, the dishes shall first be thoroughly rinsed with water
- 3) Spectrophotometer

V. REAGENTS:

- 1) **Sulfuric acid**
- 2) **Acetic acid (glacial)**
- 3) **Alkaline solution**
 - Dissolve 200 ± 2 g of sodium hydroxide (NaOH) in about 800 ml of water. Add 50 ± 0.5 g of $\text{EDTA Na}_2 \cdot 2\text{H}_2\text{O}$ ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 - 2\text{H}_2\text{O}$) and dissolve. Cool to room temperature. Then dilute into 1000 mL with water.
 - Store this solution in plastic bottle.
- 4) **Sodium azide solution (!!!! toxic !!!!)**

- Dissolve 0.05 ± 0.005 g of sodium azide (N_3Na) in 90 ml of water, and dilute to 100 ml with water.
- 5) **Sulfamic acid solution**
 - Dissolve 0.75 g of sulfamic acid (amidosulfonic acid, $\text{H}_3\text{NO}_3\text{S}$) in 1000 ml of water.
 - This is the alternative solution for sodium azide solution.
- 6) **Sodium salicylate solution**
 - Dissolve 1 ± 0.1 g of sodium salicylate ($\text{C}_6\text{H}_4\text{OHCOONa}$) in 100 ml of water.
 - Prepare this solution at the day of use.
- 7) **Nitrate (NO_3) stock solution (1000 mg N/l)**
 - Dry potassium nitrate (KNO_3) in oven at 105°C for 2 hrs. Take 7.215 ± 0.001 g of KNO_3 , dissolve with water, transfer to 1000 mL volumetric flask and fill up to the marked line with water.
 - This concentration is "not" nitrate (NO_3), this is the concentration of nitrogen (N).
- 8) **Nitrate standard solution (1 mg N /l)**
 - Prepare this solution at the day of use.

VI. PROCEDURE:

Calibration

- Take 0, 1, 2, 3, 4 and 5 ml of 1mgN/l standard solution to a series of 50 ml evaporation dishes. Follow the procedure of the measurement. Measure absorbance with spectrophotometer and plot the calibration curve, amount vs. absorbance.

Blank test

5 ml of water is preceded in the same manner.

Measurement

- 1) The maximum test portion volume which can be used for the determination of nitrate concentration up to $\text{N} = 0,2$ mg/l is 25 ml. Use smaller test portions as appropriate in order to accommodate higher nitrate concentrations. Before taking the test portion, allow laboratory samples containing suspended matter to settle centrifuge them or filter them through a washed glass fiber filter paper. Neutralize samples having a pH value greater than 8 with acetic acid (2) before taking the test portion.
- 2) Add 0,5 ml of sodium azide solution (4), and 0,2 ml of acetic acid (2).
- 3) Wait for at least 5 min, and then evaporate the mixture to dryness in the boiling water bath.
- 4) Add 1 ml of sodium salicylate solution (5), mix well and evaporate the mixture to dryness again.
- 5) Remove the dish from the water bath and allow the dish to cool to room temperature.
- 6) Add 1 ml of sulfuric acid (1) and dissolve the residue in the dish by gentle agitation. Allow the mixture to stand for about 10 min.
- 7) Then add $10 \text{ ml} \pm 0,1$ ml of water followed by 10 ml of alkali solution (3).
- 8) Quantitatively transfer the mixture to a 25 ml one-mark volumetric flask, but do not make up to the mark. Place the flask for $10 \text{ min} \pm 2$ min. Then, make up to the mark with water.
- 9) Measure the absorbance of the solution at 415 nm in cells of optical path length 40 mm or 50 mm against distilled water as a reference. Tests have indicated that the absorbance of the colored solutions remains constant for at least 24 h.

Flow of the Measurement

sample solution maximum 25 mL ---> evaporation dish (50 mL)
| start blank test in this step (use 5 mL of water instead of sample solution)
|
| <--- sulfamic acid solution (or sodium azide solution) 0.5 mL
| <--- acetic acid 0.2 mL
leave for 5 min. or more
|
heat heat to dryness
| water bath
|
cool to room temperature
|
| <--- salicylic acid solution 1 mL
mix
|
heat heat to dryness
| water bath
|
cool to room temperature
|
| <--- H₂SO₄ 1 mL
dissolve
|
leave for 10 min.
| <--- water 10 mL
| <--- alkaline solution 10 mL
transfer to 25 mL volumetric flask
|
leave at 25 C, 10 ± 2 min.
|
fill up <--- water up to 25 mL
|
Measure spectrophotometer
 415 nm
 cell: 40 to 50 mm
 reference: water

VII. EXPRESION OF REAULTS

Calculation

Calculate the actual NO₃-N using the equation:

$$\text{Nitrate (mg NO}_3\text{ /L)} = (\text{Ms} - \text{Mb})/\text{Vp}$$

where,

Ms: amount of nitrate in sample solution (mg nitrogen)

Mb: amount of nitrate in blank (mg nitrogen)

Vp: volume of the aliquot of sample solution taken (mL)

Conversion table

Relationship between nitrate as nitrogen (NO₃-N) and nitrate ion (NO₃⁻) is as follows.

Table. Conversion table

	mg NO ₃ / L	mg N / L
1 mg NO ₃ / L	1	0.226
1 mg N / L	4.427	1

- Report the result to one decimal place, and two digit significant figures.
ex. 1.767mg/l rounded off to two decimal place is 1.8mg/l.

13 Determination of Kjeldahl Nitrogen

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- ISO 5663:1984 Water quality - Determination of Kjeldahl nitrogen - Method after mineralization with selenium

Type of sample and Range

- The method is applicable in the range 0-200 mg/l

II. PRINCIPLE

- The term 'total Kjeldahl nitrogen' refers to the combination of ammonia and organic nitrogen. However, only the organic nitrogen compounds appearing as originally bound nitrogen are determined in this test. Nitrogen in this form is converted into ammonium salts by sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a nessler method modified for use with highly acidic samples resulting from the digestion.

III. SAMPLING AND SAMPLE PRESERVATION

- Laboratory samples shall be collected in polyethylene or glass bottles. They should be analyzed as quickly as possible, or else stored at between 2 and 5°C until analyzed. Acidification with sulfuric acid (3) to < pH 2 may also be used as an aid to preservation, provided that possible contamination of the acidified sample by absorption of any atmospheric ammonia is avoided.

IV. APPARATUS

- 1) Kjeldahl digester
- 2) Boiling chips
- 3) Pipette
- 4) Pipette filler
- 5) Safety glasses
- 6) Safety shield
- 7) Goggles, finger
- 8) Cylinder graduated
- 9) mixing tall form 25 ml .

V. REAGENTS

- 1) Hydrogen peroxide, 50 %, Potassium Hydroxide solution, 8 N, Sulfuric Acid, AR, Mineral stabilizer Cat. No. 23766-26, Nessler's Reagent, Extra alkaline Cat No. 21194-49, Polyvinyl Alcohol Dispensing Agent Cat No. 23765-26 and TKN Indicator Cat. No. 22519-26
- 2) Preparation of ammonium chloride stock solution (1000 mg/l N = 1220 mg/ NH₃) - Dissolve 3.819 g anhydrous NH₄Cl (dried at 100°C) in water and dilute to 1000 ml. 1ml = 1.00 mg N
- 3) Preparation of standard – Prepare 1.0 mg/l standard by diluting (a) above with de-ionized water.

VI. PROCEDURE:

Digestion

- A two-phase digestion process is employed.
- Dehydration and charring of the sample using sulfuric acid. In this process, the organic nitrogen is reduced to ammonia.

- Completion of sample decomposition using hydrogen peroxide. In this process, the carbonaceous material is oxidized.
- 1) Add 3 ml of sulfuric Acid and boiling chips to flask
- 2) Turn water to aspirator on. Set heater temperature dial to 440 °C.
- 3) Place flask weight, then fractionating column on flask. Place flask on heater
- 4) Heat until acid is refluxing or white acid vapors are present
- 5) Allow the sample to char for 3-5 minutes.
- 6) Add 10 ml of 50% hydrogen peroxide
- 7) Boil off excess hydrogen by heating for 2 more minutes.
- 8) Remove the manifold from the digestion flask. Take the flask off the heater. Allow the flask to cool.
- 9) Cool to room temperature, then dilute to mark with deionized water. Invert several times to mix. The digest is ready for colorimetric analysis unless it is turbid.
- 10) If turbid filter then proceed to colorimetric analysis.

Colorimetric analysis

- 1) Pipette an appropriate analysis volume (10 ml) into a graduated cylinder.
- 2) Add one drop of TKN Indicator
- 3) Add 8 N KOH Standard Solution, one drop at a time, swirling between each addition, until the first flash of pale blue appears (pH 3).
- 4) Add three drops of mineral stabilizer. Stopper. Invert several times to mix.
- 5) Add three drops of Polyvinyl alcohol. Stopper. Invert several times to mix.
- 6) Fill each cylinder to the 25 ml mark. Stopper. Invert several times to mix.
- 7) Add one 1 ml of Nessler's Reagent. Stopper. Invert several times to mix.
- 8) Using deionized water treated in the same way, place sample in sample compartment, zero the spectrophotometer set by pressing 42 then stored programme and setting wavelength at 460 nm (in the concentration mode) then press zero button. Remove blank and place sample then read the mg/l Kjeldahl nitrogen (N) from the display. The standard solution should read 33 mg/l TKN in this step.

VII. EXPRESION OF REAULTS

Calculation

Calculate the actual Total Kjeldahl Nitrogen using the equation:

$$\text{Kjeldahl-N(mg/l)} = 75 \times A / B \times C,$$

where

A = mg/l reading displayed

B = ml of sample digested

C = ml digest taken for analysis

14 Determination of Phosphorous Phosphate

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- (ISO 6878:2004) Water quality - Determination of phosphorus - Ammonium molybdate spectrometric method

Type of sample and Range

- The methods are applicable to all kinds of water including seawater and effluents. Phosphorus concentrations within the range of 0,005 mg/l to 0,8 mg/l may be determined in such samples without dilution.

II. PRINCIPLE

- Orthophosphate reacts with ammonium molybdate to form molybdophosphoric acid. This is transformed by the reductant (ascorbic acid) to the intensely colored complex known as molybdenum blue. Addition of potassium antimonyl tartrate increases the coloration and the reaction velocity at room temperature.

III. SAMPLING AND SAMPLE PRESERVATION

- Collect the laboratory samples in polyethene, polyvinylchloride or preferably glass bottles. In the case of low phosphate concentrations, use glass bottles.
- The use of sampling bottles with cap lines should be avoided as these may contain phosphorus.

IV. APPARATUS:

- 1) Acid washed glassware.
- 2) Spectrophotometer for use at 880 nm

V. REAGENTS

- 1) Reagents: 5N H₂SO₄.
- 2) Ammonium molybdate (20g/500 ml)
- 3) Ascorbic acid Soln. (1.32g/75 ml)
- 4) Antimonyl potassium tartrate soln. (0.2743 g/100 ml)
- 5) Mixed Reagent: Mix the above four reagents solution at the volume ratio of 10 : 3 : 6 : 1
- 6) De-ionized water
- 7) Stock phosphate solution (4.390 g of KH₂PO₄/L)
- 8) Working solution – Dilute 10 ml of the stock solution to 100 ml and mix well. Then dilute 10 ml of this solution to 1 liter and mix.
- 9) Standards

Volume of working phosphate solution	Concentration when diluted to 40 ml
0	0.0
1.0	0.025
2.0	0.050
3.0	0.075
4.0	0.1
8.0	0.2
12.0	0.3
16.0	0.4

VI. PROCEDURE:

- 1) Take 50 ml of filtered sample, blank & standard in dry test tube.
- 2) Adjusting pH to 5-9 with dil. NaOH. Add a drop of phenolphthalein.
- 3) If red color develops, add H₂SO₄.
- 4) Add 8 ml combined and mix.
- 5) Wait for 10 minutes
- 6) Measure absorbance at 880nm. and from the graph, read the phosphorus concentration in the sample.

Accuracy check

- Using the 50 mg/l as PO₄, add 0.1, 0.2, 0.3 ml of standard to 25 ml sample. Mix thoroughly. Analyze spiked samples and compare the results with original sample. The phosphate concentration should increase 0.2 mg/l for each 0.1 ml of standard added.

VII. EXPRESION OF REAULTS

Calculation

Calculate the actual PO₄-P using the equation:

$$\text{Phosphate (mg P /L)} = C_s - C_b$$

where,

C_s: concentration of phosphate in sample solution (mg P/l)

C_b: concentration of phosphate in blank (mg P/l)

Conversion table

Relationship between phosphate as phosphorous (PO₄-P) and phosphate ion (PO₄⁻) is as follows.

Table. Conversion table

	mg PO ₄ -P / L	mg PO ₄ ⁻ / L
1 mg PO ₄ -P / L	1	3.07
1 mg PO ₄ ⁻ / L	0.326	1

- Report the result to two decimal place, and two digit significant figures.
ex. 0.767mg/l rounded off to two decimal place is 0.77mg/l.

15 Determination of Oil and Grease

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- This procedure describes the procedure of oil and grease analysis of aqueous sample following US-EPA method 1664.

Type of sample and Range

- This method is for determination of n-hexane extractable material (HEM)
- This method is not applicable to measurement of materials that volatilize at temperatures below approximately 85°C.
- This method is capable of measuring HEM in the range of 5 to 1000 mg/L, and may be extended to higher levels by analysis of a smaller sample volume collected separately.
- For this method, the Method Detection Limit (MDL) has been determined as 1.4 mg/L for HEM..

Interferences

- Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts that affect results. Specific selection of reagents and purification of solvents may be required.
- Glassware is cleaned by washing in hot water containing detergent, rinsing with tap and distilled water, and rinsing with solvent or baking. Boiling flasks that will contain the extracted residue are dried in an oven at 105–115°C and stored in a desiccator.

II. PRINCIPLE

- A 1-L sample is acidified to pH <2 and serially extracted three times with n-hexane in a separatory funnel. The extract is dried over sodium sulfate. The solvent is evaporated from the extract and the HEM is weighed. Quality is assured through calibration and testing of the extraction, concentration, and gravimetric systems.

III. SAMPLING AND SAMPLE PRESERVATION

- Collect approximately one (1) liter of sample in container.
- Sample bottle must "NOT" be pre-rinsed with sample before collection.
- pH adjustment: pH<2 with HCl(1+1) or H₂SO₄(1+1)
- Required amount of acid must be determined prior to sampling.
- Don't dip pH electrode, pH test paper or glass rod into the sample in sample bottle

IV. APPARATUS

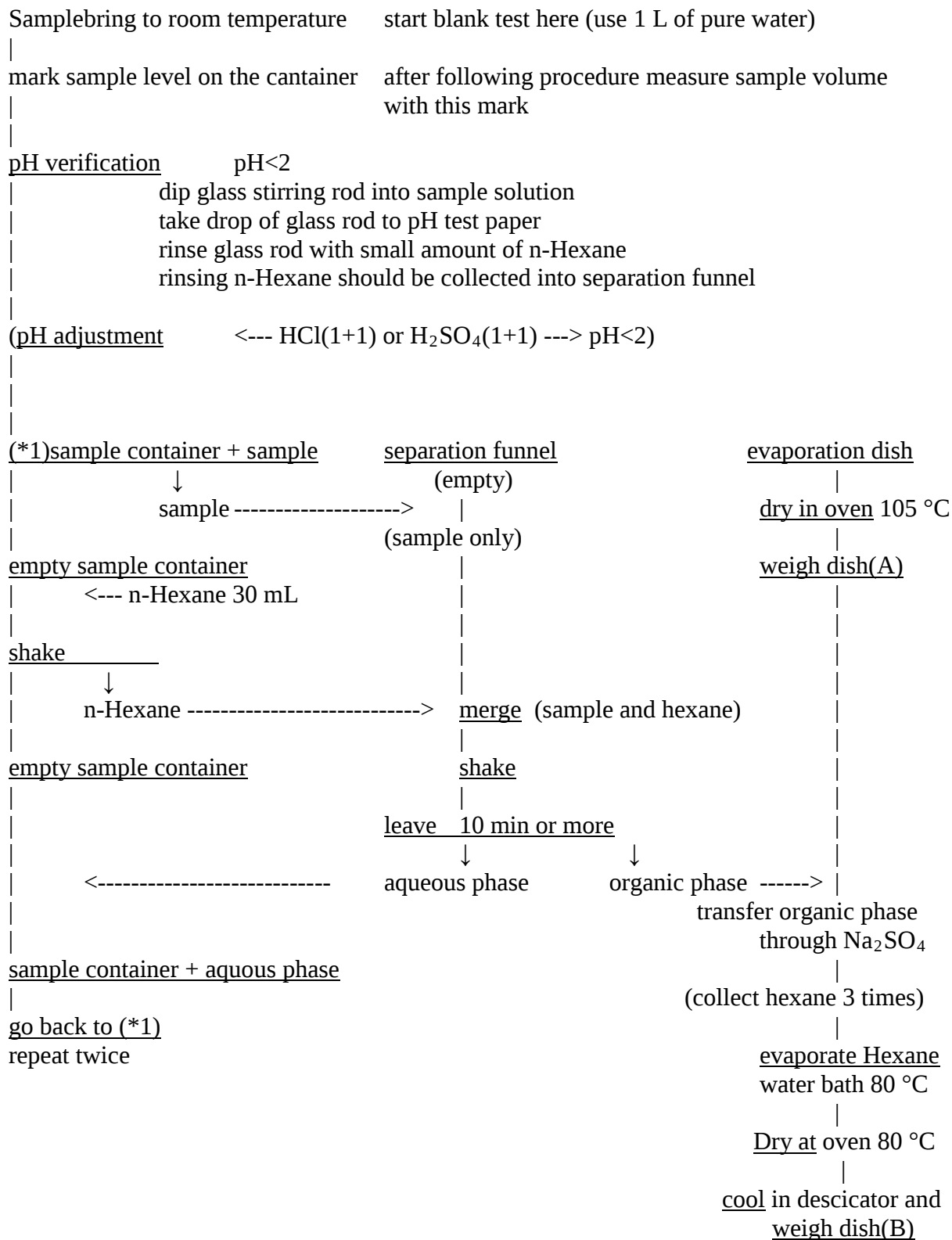
- 1) Sampling container
- 2) Glass
- 3) Cleaning of sampling container
- 4) wash with detergent and rinse with tap water
(when wash with solvent, "be careful of fire ")
- 5) heat in drying oven at 200 to 250 °C
- 6) Separatory funnel
- 7) glass, 1000 mL, PTFE stopcock
- 8) Centrifuge
- 9) Water bath
- 10) Drying oven

V. REAGENTS

- 1) Hydrochloric acid (1+1) or sulfuric acid (1+1)
- 2) n-Hexane
- 3) 85% purity, 99% minimum saturated C6 isomers, residue less than 1 mg/L
- 4) Acetone
- 5) Sodium sulfate anhydrous (Na_2SO_4)
- 6) Granular

VI. PROCEDURE:

Flow chart of measurement



Blank test

- Blank test should be carried out with each analytical batch. Water should be used instead of sample solution. If blank test result is greater than the minimum level (5 mg/L), analysis of samples is halted until the source of contamination is eliminated and blank shows no evidence of contamination.

VII. EXPRESSION OF RESULTS

Calculation

Oil and Grease (mg/L) = (B - A) / volume

B: weight of evaporation dish after evaporation (mg)

A: weight of evaporation dish before evaporation (mg)

volume: sample volume (L)

16 Determination of Phenol

I. SCOPE AND GENERAL DISCUSSION

Applied Method

- TCVN 6216-1996(ISO6439-1-1990), Water quality - Determination of phenol index. 4-aminoantipyrine spectrometric method after distillation

Type of sample and Range

- This is the method for determining the phenol index in drinking waters, surface water and waste water.
- After a preliminary distillation, the test samples are analyzed by direct colorimetric method. This method is capable of measuring the phenol index in test samples that contain more than 0.10 mg/l in the aqueous phase(without chloroform extraction), using phenol as a standard.
- When the lower MDL is required, the chloroform extraction method is capable of measuring the phenol index without dilution from about 0.002 mg/l to about 0.1 mg/l when the coloured end-product is extracted and concentrated in chloroform phase, using phenol as a standard.
- In this SOP, only the direct colorimetric method is described. The MQL(Method Quantification Limit): 0.10 mg/L , 0.002mg/L (with chloroform extraction).

Interferences

- Common interferences that may occur in waters are phenol –decomposing bacteria, oxidizing and reducing substances, and strongly alkaline conditions of the sample. Biological degradation is inhibited by the addition of copper(III) sulfate to the sample. Furthermore, some methods for eliminating certain interferences are suggested in ISO6439-1, 1990.

II. PRINCIPLE

- The term “phenolic compounds ” means hydroxyl derivatives of benzene and its analogues. On the other hand, the term “phenol index” as used in ISO only includes phenols which react with 4-aminoantipyrine under the conditions specified to give colored compounds.
- In a water containing phenol itself, there will usually be associated with it other phenolic compounds whose sensitivity to the reagents used in the following methods may not necessarily be the same.
- The percentage composition of the various phenolic compounds present in a given test sample is unpredictable. It is obvious, therefore, that a standard containing a mixture of phenolic compounds cannot be made applicable to all test samples. For this reason, phenol(C_6H_5OH) has been selected as a standard, and any colour produced by the reaction of other phenolic compounds is measured as phenol and reported as the phenol index.
- Separation of phenolic compounds from impurities and preservative agents by distillation. The rate of volatilization of the phenolic compounds is gradual, so that the volume of the distillate must equal that of the test sample being distilled.
- Reaction of the steam-distillable phenolic compounds with 4-aminoantipyrine at a pH of 10.0 ± 0.2 in the presence of potassium hexacyanogerrate(III) to form a coloured antipyrine dye.
- Measurement of the absorbance of the dye at 510nm. The phenol index is expressed as milligrams of phenol(C_6H_5OH) per litre.

III. SAMPLING AND SAMPLE PRESERVATION

- Container : Glass
- Minimum Sample Size:500mL-1L
- Phenolic compounds in water are subject to both chemical and biochemical oxidation. Therefore , unless the samples are analyzed within 4 hour of collection , the shall be preserved when collected, using the following procedure;
 - a) acidify the samples to a pH of approximately 4.0 with phosphoric acid . use methy orange or a pH meter to check the pH.
 - b) inhibit biochemical oxidation of phenolic compounds in the sample by adding 1.0 g of copper (III) sulfate per litre of the samply;
 - c) store the sample in the cold (5 °C to 10°C) and analyze the preserved samples within 24 hour of collection.

IV. APPARATUS

- 7) Distillation apparatus; all glass, consisting of a 1 liter borosilicate glass distilling apparatus with Graham condenser or equivalent.
- 8) pH meter; and suitable electrodes
- 9) Spectrometer

V. REAGENTS

- 10) 4-aminoantipyrine solution
 - dissolve 2 g of 4-aminoantipyrine($C_{11}H_{13}N_3O$) into water, and dilute to 100 ml with water
 - prepare this solution just before use
- 11) Ammonium chloride solution (20 g/L)
- 12) Ammonium hydroxide (solution)
- 13) Potassium sodium tartrate solution (buffer solution, pH=10)
 - dissolve 34 g of ammonium chloride (NH_4Cl) and 200g of potassium sodium tartrate ($KNaC_4H_4O_6$) in 700 ml of water. And add 150 ml of ammonium hydroxide solution and dilute to 1000 mL with water.
- 14) $CuSO_4$ solution
 - dissolve 190 g of copper (II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in water and dilute with water to 1000 ml
- 15) Hydrochloric acid
- 16) Methyl orange solution (indicator)
 - dissolve 0.5 g of methyl orange in water and dilute to 1000mL with water
- 17) Phenol stock solution (1 g/L)
 - dissolve 1.00 g of phenol in water (*) into 1000 ml volumetric flask, and dilute to the marked line.
 - (*) use freshly boiled and cooled water

this solution is stable in one week.
- 18) Phenol standard solution (0.01 g/L)
 - take 10 ml of stock solution (1 g/L)(8) into 1000 ml volumetric flask and dilute to the marked line with water (*)
 - (*) use freshly boiled and cooled water
 - prepare this solution on the day of use
- 19) Phenol standard solution (0.001 g/L)

- take 50 ml of standard solution (0.01 g/L)(9) into 500 ml volumetric flask and dilute to the marked line with water (*)
 - (*) use freshly boiled and cooled water
- prepare this solution on the day of use
- 20) Phosphoric acid (1+9)
- mix one volume of phosphoric acid with nine volume of water
- 21) Potassium hexacyanoferrate (III) solution (80 g/L)
- dissolve 8.0 g of potassium hexacyanoferrate(III) in water and dilute to 100 ml with water.
 - filter if necessary
 - this solution is stable in one week.
- 22) Sodium sulfate undyrous (NaSO_4)

VI. PROCEDURE:

Flow chart of preservation

```

sample ←  $\text{H}_3\text{PO}_4$  → pH 4
|
|   use pH meter or methyl orange
|   ←  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  (1 g per 1000 mL sample)
|

```

(distillation)

```

aliquot of sample           500mL
|
|   transfer                 to beaker (1000 mL)
|
|   pH adjust      ←  $\text{H}_3\text{PO}_4$  → pH 1 ~ 2
|
|   transfer                 to distillation flask
|
|   connect receiving cylinder      500 mL measuring cylinder
|
|   distillation    until 400 mL is distilled
|
|   stop distillation      leave for cooling
|
|   add water to distillation flask ← 100 mL water
|
|   distillation
|
|   stop distillation      distillate becomes 500 mL
|

```

Flow chart of color forming

aliquot of distillate 100 mL
 |
transfer to beaker (250 ml)
 | ← 5 mL buffer solution (Potassium sodium tartrate solution)
 |
adjust pH ← ammonium hydroxide → pH 9.8 ~ 10.2
 | ← 2 ml 4-aminoantipyrine solution
mix immediately
 | ← 2 mL potassium hexacyanoferrate (III) solution
 |
mix immediately
 |
leave for 15 min
 |
measure spectrophotometer 510 nm
 reference: water

Calibration curve

- Take 0, 50, 100 and 200 ml of phenol standard solution (0.01 g/L) respectively into a series of 500 ml beakers. Add 5 mL of copper sulfate (CuSO₄) solution to all flasks. Dilute to 500 mL with water.
- Apply distillation process and color forming process to these solutions respectively according to the manner in section 2.
- Plot concentration versus absorption curve, and prepare calibration curve.

Blank test

- Proceed all process described in section 2. Use pure water instead of sample solution.

VII. EXPRESION OF REAULTS

- Estimate the phenol index of the test portion by reference to the calibration graph and to the absorbance obtained with the solution of sample.

$$m/V_0 \times 1000$$

where;

m : the mass, in milligrams, of phenol equivalent to the phenolic compounds in the test portion

V₀ : volume, in ,milliliters, of the test portion

APPENDIX-3

HANDBOOK FOR

STATISTICAL ANALYSIS OF WATER QUALITY DATA

SCOWEM

Project for Strengthening Capacity of Water Environmental Management in Vietnam

Output 2-1: Water Quality Monitoring

Handbook for Statistical Analysis of Water Quality Data

October 2012

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Appendices

Appendix 1: Frequently used Excel Functions

Appendix 2: Statistical Look-up Tables used in the Tests

Appendix 3: Process Flow Charts.

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Abbreviations

Abbreviation	Meaning
$\hat{\lambda}$	Mean and variance correction factor in Cohen's Procedure
$\bar{x}$	Mean of a set of values
$\bar{x}_d$	The mean of the values in a data array which are above the DL or RL
<	Less than
>	Greater than
$\leq$	Equal to or less than
$\geq$	Equal to or greater than
a	Lower limit value for w/s in Studentized Range Test
b	Upper limit value for w/s in Studentized Range Test
C	Calculated statistic in Dixon's Extreme Value Test
DL	Detection Limit
DQO	Data Quality Objectives
h	Proportion of non-detects in Cohen's Adjustment Procedure
m	The number of samples in a data array which are higher than the DL or RL
n	The number of values in an data array
RL	Reporting Limit
s	Standard deviation of a given set of values
s^2	Variance of a given set of values
s_d^2	The variance of the values in a data array which are above the DL or RL
t	The t statistic defining the number standard deviations corresponding to a particular probability level
USEPA	United States Environmental Protection Agency
w	Range of a set of values (Studentized Range Test)
$X_{(n)}$	The nth value in an array of values of X
α	
μ	Mean of a set of values
σ	Standard deviation of a given set of values
σ^2	Variance of a given set of values
Y	Parameter relating s_d^2 , $\bar{x}_d$ and DL in Cohen's Procedure
CV	Coefficient of Variation
SSS	Sample sum of squares
SAD	Sum of absolute differences
a	Geary's test statistic
Z	
$X'_{(i)}$	The first transformation of the ith variable X
$X''_{(i)}$	The second transformation of the ith variable X
k	The reflection constant applied to a negatively skewed data series
p	
r	The Pearson correlation coefficient
R^2	The square of the Pearson correlation coefficient
nd	Not detected
np	Not present
dof	Degrees of freedom

1 Introduction

1.1 Purpose of this Handbook

This handbook has been prepared following a series of two-day training sessions in the Provinces of Hanoi, Hai Phong, Hue, Ho Chi Minh City and Baria Vung Tau. The purpose of the training was to introduce the staff of the respective DONRE departments responsible for environmental monitoring to some of the basic statistical techniques. This would improve the quality of the environmental data on which decisions were being made and to provide measures of the confidence around the conclusions drawn from those data.

Previous training under Topic 2-1 has covered the structured approach to the design of water quality monitoring plans based on the Data Quality Objectives (DQO) process developed by the USEPA (USEPA, 1994). That training had introduced the requirement for statistical robustness in the design and implementation of monitoring plans (SCOWEM, 2011a) and a critical examination of the design of the current water quality plan in the respective DONRE (SCOWEM, 2011b). This approach was aimed at strengthening the capacity within the various DONRE to design their water quality monitoring plans in-house, or for those DONRE where the preparation was carried out by a third party to review the design from a point of knowledge. Such capacity would ensure that the data that are generated by the monitoring process are of the highest quality and fit for the purposes of the monitoring plan.

A third training (SCOWEM, 2012) provided the DONRE staff with practical experience in carrying out basic data screening and statistical examination of both example data sets and also using data collected as part of their own monitoring program.

This handbook was specifically written following the third training in order to address questions and comments that arose during the course. It is intended that it be used in conjunction with the training handout and provides the reader with a step-by-step guide to the use of techniques to:

- Identify extreme and outlier values.
- To maximize information where measurements indicate that environmental concentrations are below the limit of detection by the analytical method being used.
- To determine the confidence level of measured water quality meeting, or failing to meet, the relevant environmental water quality standard.
- To determine whether concentrations from two different locations or measured at two different times are different at a given level of confidence.
- To determine whether a trend in concentration with respect to distance or time is significant at a given level of confidence.

1.2 Flow Charts

Six flow charts are provided in Appendix 3 which take the user through the processes of

1. Checking incoming data
2. Managing non-detects
3. Testing for normality of data
4. Testing for compliance with a standard

5. Testing for differences between two sets of data
6. Linear trend analysis

These are intended to provide the user with a checklist of the individual tasks to be completed while carrying out particular process associated with the management and analysis of data associated with water quality monitoring plans.

1.3 Reference Material

Many textbooks have been written on the topic of environmental statistics, which assume a previous knowledge of statistics by the reader. This handbook does not seek to replace such textbooks. It is the intention that the handbook acts more as an annotated series of check lists or recipes which the user can use during their normal day to day conduct of data handling and data interpretation.

A bibliography provides details of some books and links to web sites which will provide additional information which may not be in the Handbook.

As part of the development of the DQO process the USEPA produced a comprehensive manual (USEPA, 1998) containing a wide range of data analysis techniques that are appropriate at all levels of data analysis. This Handbook will at times refer to sections the USEPA manual. It is hoped that the user of this handbook will, when confident in the procedures described in the handbook, explore the manual and discover new ways of presenting and analyzing the data collected during the monitoring programs. Hard and soft copies of the manual were left with each of the DONRE departments taking part in the third training.

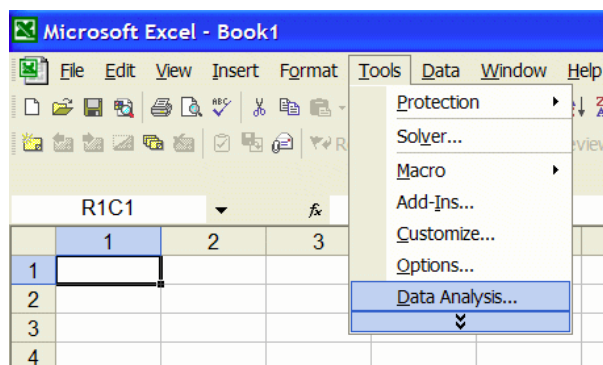
1.4 Software Requirements

1.4.1 PC users operating with Microsoft Windows.

Users who have access to a computer on which has been installed Microsoft® Excel® should ensure that the 'add in' Data Analysis ToolPak has been activated. The tool pack is used extensively when plotting histograms, carrying out F-tests to check for equality of variances and for carrying out t-test for comparison of means.

ToolPak is accessed through Data Analysis located in the Tools dropdown box as shown in Figure 1.1.

Figure 1.1. Access to the Data Analysis tool pack.

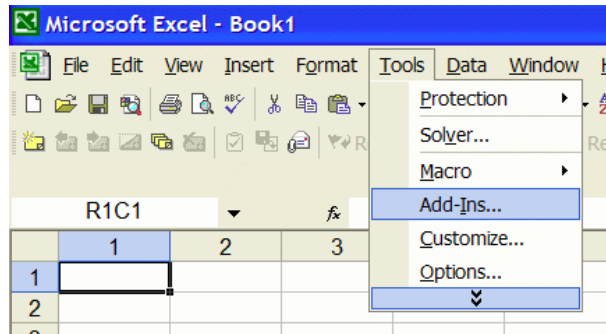


1.4.1.1 Installation of the Data Analysis ToolPak

If Data Analysis is not displayed in the Tools menu it can be installed as described below.

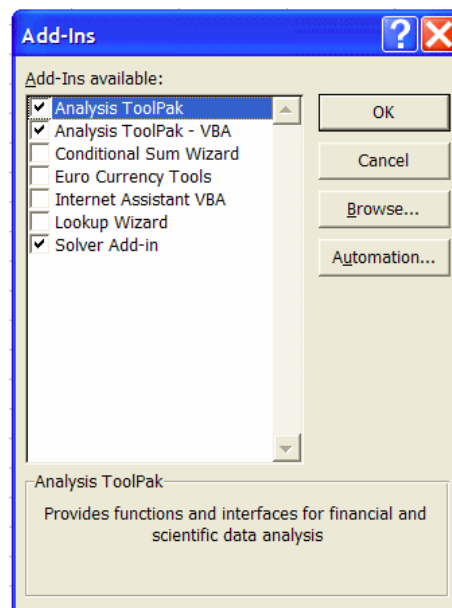
Locate Add-Ins... in the tools menu as shown in Figure 1.2

Figure 1.2 Location of Add-Ins function in the Tools menu



Clicking on Add-Ins... will activate the selection box, Figure 1.3, from where ticking the Analysis ToolPak option, and clicking OK will install the data analysis tools on the Tools menu.

Figure 1.3. Options within the Add-Ins... dropdown.



1.4.2 PC users operating under Mac OS X

User who have access to a Mac computer on which has been installed a pre year-2008 Microsoft Office for Mac system will be able to activate the Data Analysis ToolPak. Those with more recent version of Office will find that the Data Analysis Add In is no longer available.

However, a free down load of an almost identical tool-pack lookalike, StatPlus:mac LE, can be obtained from <http://www.analystsoft.com> . The LE version contains all of the routines contained in the original Excel Took-pack.

2 The Requirement for Statistical Analysis

The environment is not a perfectly ordered system and contains a large element of randomness. Measurements of the concentration of a particular contaminant in one hundred samples of water taken from the same location in a river or lake over a period of time will reveal a range of concentrations. Such sampling was simulated during Training Session 2 when an artificial river containing several hundred samples in the form of squares of paper on which was printed a concentration. The samples were generated from a normal population with a known mean and standard deviation. The exercise demonstrated the possible variability between individual single samples and the benefits in taking and measuring replicate samples and the benefit of making composite samples.

In the field the range of the concentrations measured will depend to a large extent on the time period over which the one hundred samples were taken. Generally the longer the time period over which samples were taken the greater will be the range of concentrations.

It is the uncertainty around the variability and randomness of sampling which requires a statistical approach to be taken. It is through the statistical approach that the random uncertainty is quantified and a level of confidence assigned to any conclusion based on the data.

3 The Basis of the Statistical Tests

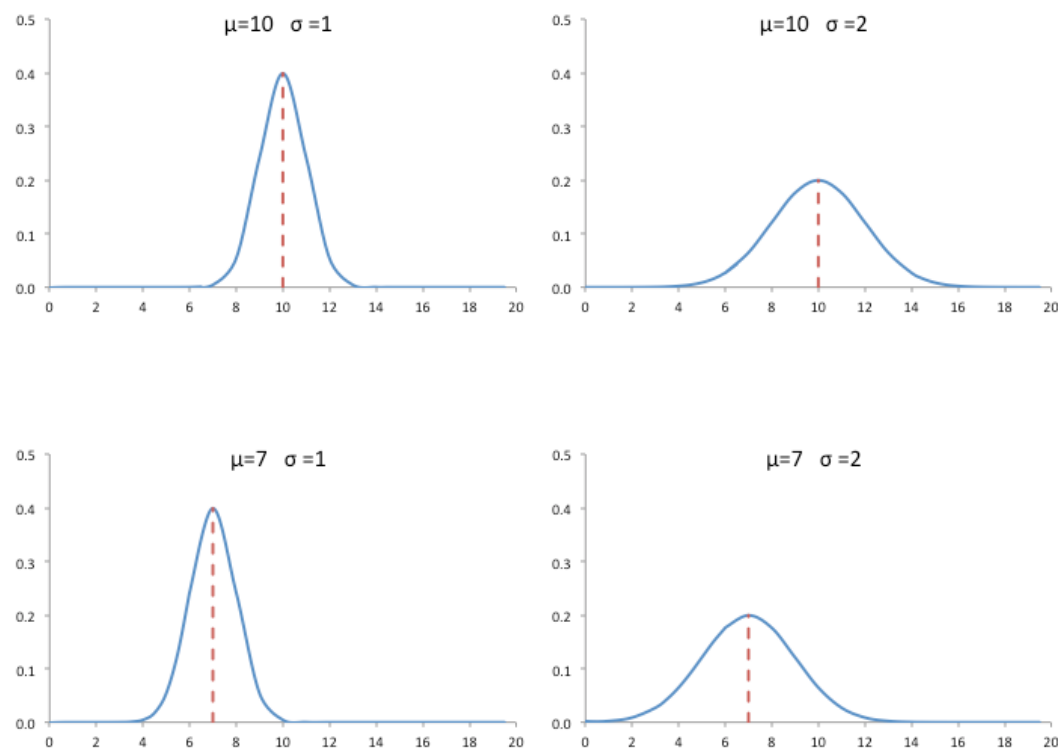
3.1 The Normal distribution

The normal distribution is a very important class of statistical distributions. All normal distributions are symmetric and have bell-shaped density curves with a single peak.

To speak specifically of any normal distribution, two parameters have to be quantified: the mean, where the peak of the density occurs, and the standard deviation, which indicates the spread of the bell curve. The Greek symbol μ (mu) is used to represent the mean and the symbol σ (sigma) to indicate the standard deviation.

Different values of μ and σ yield different normal density curves and hence different normal distributions. Drawing 3.1 shows the shape of the normal density curve for different values of μ and σ .

Drawing 3.1. Effect of different values of μ and σ on the location and shape of a normal curve.



The drawing clearly shows that changing the value of μ while leaving σ unchanged simply moves the mid point of the curve along the number line while the shape remains the same. Keeping μ constant and changing σ keeps the position the same while the shape changes. Changing both μ and σ results in the position of the mid point of the curve being changed and also the shape is changed. In all cases the area between the curve and the number line, the x-axis remains constant.

All normal density curves satisfy the following properties, irrespective of the mean concentration and the standard deviation.

- i. 68% of the observations fall within 1 standard deviation of the mean, that is, between $\mu - 1\sigma$ and $\mu + 1\sigma$.
- ii. 95% of the observations fall within 2 standard deviations of the mean, that is, between $\mu - 2\sigma$ and $\mu + 2\sigma$.
- iii. 99.7% of the observations fall within 3 standard deviations of the mean, that is, between $\mu - 3\sigma$ and $\mu + 3\sigma$.

Thus, for a normally distributed water quality determinand, almost all values will lie within three standard deviations of the mean and up to three samples in every thousand falling outside that range would not be a cause for concern.

All normal density curves satisfy the property described above and this property is the basis of all the statistical tests and assumptions which are described in this handbook.

3.2 Significance Levels

Significance levels also known as levels of confidence, represent the level of confidence with which we wish to make a statement that there is a difference in water quality at two stations or that the water quality meets the required water quality standard. This is usually expressed in terms of percentage confidence, and in the case of environmental monitoring this will usually be 90% or 95%.

Statistical look-up tables represent the percentage confidence in terms of probability, denoted by the symbol α , where $\alpha = 1 - \%/100$. For example if the confidence level is 95% then $\alpha = 0.05$.

3.3 Null and Alternative Hypotheses

3.3.1 Purpose of establishing the hypotheses

When carrying out statistical analyses, tests are carried out to accept the null hypothesis (H_0) at a given level of confidence. If the null hypothesis is rejected then the alternative hypothesis (H_A) is accepted.

Typically when carrying out water quality monitoring the null and alternative hypotheses can take different forms:

3.3.1.1 Comparing with a water quality standard.

When comparing a measured concentration with the environmental standard the objective is to determine whether, for compliance, the measured concentration is, in most cases, below an upper limit defined by the environmental standard concentration.

The exception to this is the case for dissolved oxygen when the objective is to determine whether, for compliance, the measured concentration is above than the environmental standard concentration.

H_0 : The measured concentration is greater than or equal to the environmental standard at a confidence level of 95%.

H_A : The measured concentration is less than the environmental standard at a confidence level of 95%.

In the case of dissolved oxygen the alternative hypothesis would be:

H_A : The measured concentration is greater than the environmental standard at a confidence level of 95%.

3.3.2 Comparing between two stations.

When comparing concentrations from two stations the objective is to determine whether:

- i. there has been a deterioration in water quality as a result of a discharge into to the river between the two stations or
- ii. water quality is improving as a result of dilution or self purification.

In both cases there is an expectation of the direction of change.

In the case of the discharge, the expectation is that the concentration at the downstream station will be higher (for oxygen, in most cases, it will be lower) than at the upstream station

H_0 : The measured concentration at the downstream station is greater than or equal to the measured concentration at the upstream station at a confidence level of 95%

H_A : The measured concentration at the downstream station is lower than the measured concentration at the upstream station at a confidence level of 95%.

3.3.3 Comparing between two times at the same station.

When comparing concentrations from two different times at the same station the objective is to determine whether there has been a deterioration or improvement in water quality during the period at the same station. This may be to determine the effect of infrastructure changes such as a new discharge to the river or the introduction a wastewater treatment plant to treat a previously raw discharge management plan. In both cases the direction of any change in concentration will be known.

The difference between the two times may be related to seasonal differences, wet season vs dry season or a shorter time period such as, in estuarine conditions high water and low water, or in the case of lakes daytime and night-time dissolved oxygen concentrations.

In most cases the direction of change will be known and the alternative hypothesis can be phrased appropriately, for example:

H_0 : The measured concentrations following the commissioning of the wastewater treatment plant is greater than or equal to that measured at the same location prior to its construction at a confidence level of 95%.

H_A : The measured concentration following the commissioning of the wastewater treatment plant is lower than that measured at the same location prior to its construction at a confidence level of 95%.

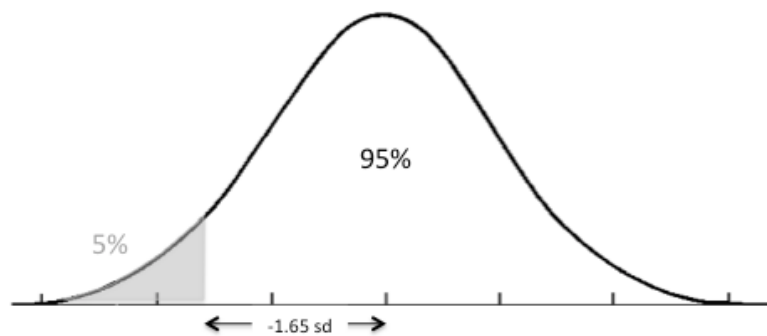
3.4 One and two tailed tests

The form of the alternative hypothesis defines whether a one-tail or a two-tail test will be applied to the data. The difference between the assumptions made regarding the two tests defines how the critical value for the test is obtained.

3.4.1 One-tailed test

The one-tailed test is used when the alternative hypothesis is that any difference that does exist can only be in one direction. Drawing 3.2 shows the area under the normal curve corresponding to 5% of the total area located on the negative side of mean; The properties of the normal curve define that this area is 1.65 standard deviations on the negative side of the mean.

Drawing 3.2. Normal distribution curve showing single-tail 95% zone and location of the single tail zone.



3.4.2 Two-tailed test

The two-tailed test is used when the alternative hypothesis is that there is a difference but that it can be a difference in either direction. Drawing 3.3 shows the area under the normal curve corresponding to two 2.5% areas, one on the positive side and one on the negative side, leaving 95% in the centre. The properties of the normal curve define that each of the 2.5% areas are 2 standard deviations either side of the mean.

Drawing 3.3 Normal distribution curve showing the two-tail 95% zone and location of each of the two tails.



3.5 Degrees of freedom

The term 'degrees of freedom', dof, will be encountered when using some look-up tables associated with different statistical tests. The number of degrees of freedom is defined as the number of values in the final calculation of a statistic that are free to vary.

For example, in order to achieve the value of 31 as the a mean of ten values (n) there is the freedom to choose any nine numbers. but the tenth number is constrained to be that number which makes the mean of the whole set to be 31. Hence there are nine ($n-1$) degrees of freedom.

Similarly, a straight line fitted through fifteen (n) pairs of numbers (x_i, y_i) is required to have a slope of 2.8 and an intercept of 0.9. Thirteen points can be located anywhere on the two-dimensional plane, the positions defined by the final two pairs of numbers are constrained in order to ensure that the slope and intercept satisfy the required values. Hence there are thirteen ($n-2$) degrees of freedom.

For each test, where the number of degrees of freedom are required for the look up table, the method of determining that number is constant and defined by the test.

4 Screening of Incoming Data

4.1 Within the expected range

When a monitoring program has been underway for more than one year there will be sufficient historic data from which to be able establish an expected range of concentrations at individual stations. These values can be used to generate a set of graphs or tables that can be used to screen any new incoming data to determine whether the values lie within the normally expected range.

The graphs or tables rely on the fact that 95% of results should lie between $\mu - 2\sigma$ and $\mu + 2\sigma$ and that 99.7% of all results should lie between $\mu - 3\sigma$ and $\mu + 3\sigma$.

The following example shows the steps in establishing a basic screening chart and table for measurements of COD at a point in a lowland river.

4.1.1 Establishment of historic data variability and normality

Tabulate the historic data and calculate the cumulative mean and cumulative standard deviation. Table 4.1 shows the dates, measured values and cumulative mean and cumulative standard deviation of the first two years of measurements.

Table 4.1. Dates and measured values of COD and the calculated cumulative mean and cumulative standard deviation.

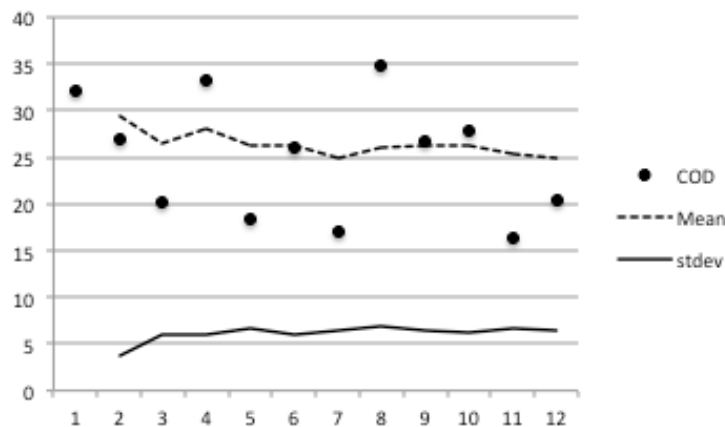
Date	COD	Mean	stdev
Feb-08	32.2		
Apr-08	26.9	29.5	3.7
Jun-08	20.3	26.4	6.0
Aug-08	33.2	28.1	5.9
Oct-08	18.5	26.2	6.7
Dec-08	26.0	26.2	6.0
Feb-09	17.0	24.9	6.5
Apr-09	34.8	26.1	7.0
Jun-09	26.7	26.2	6.5
Aug-09	27.8	26.3	6.2
Oct-09	16.3	25.4	6.6
Dec-09	20.5	25.0	6.4

The range of values and stability of the mean and standard deviation can be more readily evaluated when the information is shown graphically.

The plotted values are shown in Drawing 4.1. The data are plotted in the order that the samples were taken. For the purposes of establishing the screening chart it is only the order that the sampling and analysis were carried out and so the date information has been replaced ordinal values.

The range of concentrations is from 16.3 mg/l up to 34.8 mg/l with a mean concentration that rapidly stabilizes to around 25 mg/l. The standard deviation also stabilizes quickly at around 6.5 mg/l.

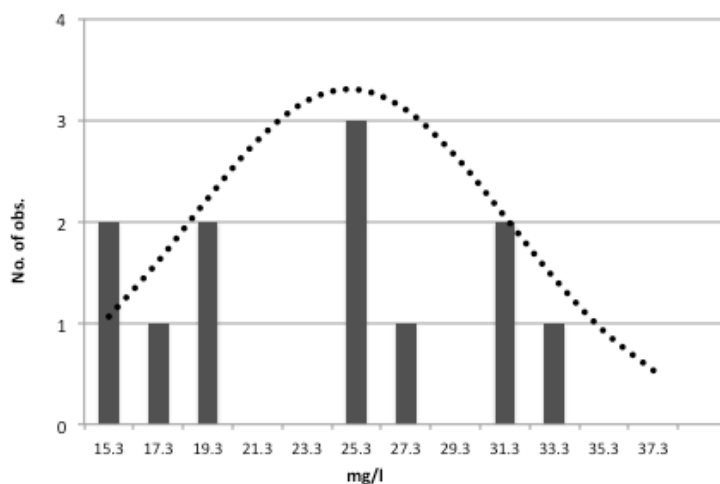
Drawing 4.1. Measured BOD Concentrations together with the cumulative mean and cumulative standard deviation.



It is also valuable to establish whether the data are normally distributed. Initially a simple histogram may be sufficient; more advanced tests for normality are described in detail in Section 6 of this Handbook.

Drawing 4.2 shows the histogram of the measured COD concentrations with the normal density curve corresponding to the mean and standard deviation of the data set. Based on a visual examination of the drawing the assumption that the data do fit a normal distribution would appear to be supported.

Drawing 4.2. Histogram of the measured COD concentrations with the corresponding normal density curve.

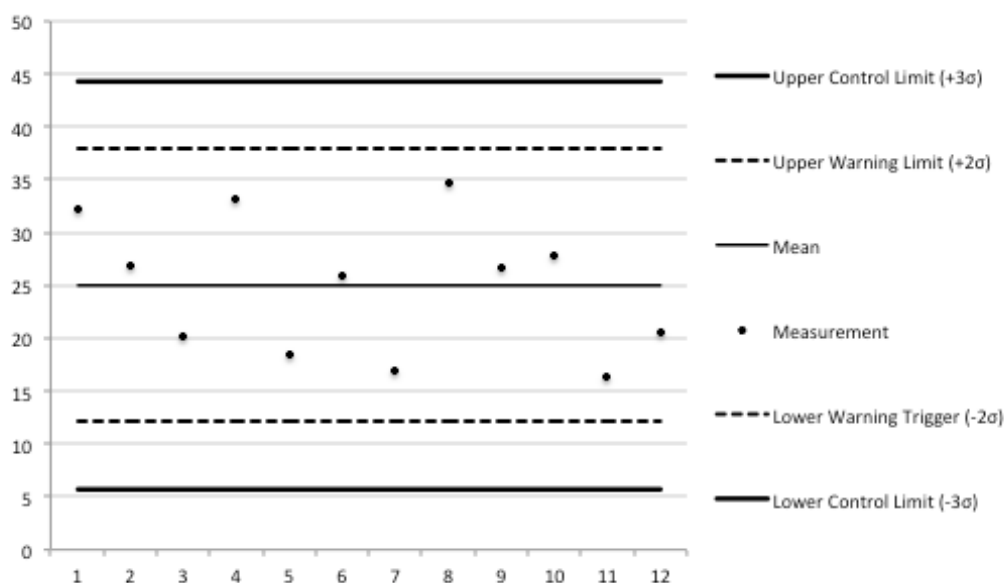


If the data are not normally distributed it may be necessary to carry out a transformation (Section 7 of the Handbook) in order to obtain normality. In such a case the mean and standard deviation should be re-calculated on the basis of the transformed data.

4.1.2 Preparation of the control chart.

Calculate $\pm 2\sigma$ and $\pm 3\sigma$ lines and plot with mean and measurements to produce the control chart for the contaminant at the particular station. This will result in a chart which resembles that shown in Drawing 4.3. The chart becomes the tool for identifying values that are outside the expected range based on the historic behavior of the determinand at that particular location.

Drawing 4.3. Control Chart for the Identification of Potential Outliers showing the 95% and 99.7 Warning and Control Tram Lines.



If there are no changes in the variability of the data it would be expected that 95% of all the data points would be located between the $\pm 2\sigma$ tram-lines, and that 99.7% between the $\pm 3\sigma$ tramlines.

A single value falling between the $+2\sigma$ and $+3\sigma$ lines or between the -2σ and -3σ lines has a 2.5% chance of occurring and should be regarded as a warning and a simple check that nothing unusual occurred during sampling or analysis. The likelihood of two consecutive values occurring in the warning zone is 0.06%. Should that occur or a single value outside the $\pm 3\sigma$ it is necessary to review all the sampling and analytical procedures unless it can be shown that the extreme values were the result of a pollution event or and extreme environmental event.

Seven or more consecutive points lying on one side of the mean ($0.57 \times 100 = 0.8\%$ chance of occurring) and indicates a shift in the mean concentration. This should trigger an investigation regarding a systematic change. This may be something as simple as changing the sampling point from the left side of the river to the right because of a change in accessibility. It may also indicate something more serious such as the introduction of a new discharge to the river that has not been notified to the monitoring group. In such a case, and the change will be permanent then a new mean and standard deviation will need to be calculated based on the data obtained since the change was implemented.

Five of six consecutive points going in the same direction indicates a significant trend in the improvement or deterioration in water quality.

4.2 Outliers

Outliers are measurements that are extremely large, or small, relative to the rest of the data and therefore may not be a true representation of the concentration in the water body from which they were collected. Outliers may occur as a result of transcription errors, data-coding errors, or measurement system problems such as a field or laboratory instrument breakdown.

However, outliers may also represent true extreme values of a distribution and indicate more variability in the environment than was expected. The act of not removing true outliers and removing false outliers both lead to a distortion of estimates of statistical parameters.

Statistical outlier tests provide statistical evidence that an extreme value does not fit in the distribution of the remainder of the data and is therefore a statistical outlier. Such tests should only be used to identify data points that require further investigation. The tests in themselves cannot determine whether a statistical outlier should be discarded, corrected or retained within a data set. Such a decision must be based on experienced based judgmental or scientific grounds.

There are five stages involved in the management of extreme values or outliers:

1. Identify extreme values that may be potential outliers;
2. Apply an appropriate statistical test;
3. Scientifically review proven statistical outliers and decide on their status;
4. Conduct data analyses with and without statistical outliers; and
5. Document all the steps taken and decisions made.

The visualisation of the data as a simple time series graphical plot may be sufficient to identify the presence of a potential outlier or outliers. Plots similar to those shown in Drawings 4.1 and 4.3 are typical form of plot effective at showing the presence of values to be investigated. If the visualisation identifies potential outliers the next step is to apply a statistical tests to confirm its status.

The statistical test proposed for testing for outliers is the Extreme Value Test or Dixon's Test.

4.2.1 Dixon's Test

Dixon's Extreme Value test can be used to test for statistical outliers when the sample size is less than or equal to 25. For data sets containing more than 25 values then the Discordance Test described in Section 4.4.4 of EPA QA/G-9 can be used but can only be used if there is only one suspected outlier in the data set.

Dixon's test considers either extreme high or extreme low values:

- i. those that are much smaller than the rest of the data (case 1) and
- ii. those that are much larger than the rest of the data (case 2).

Dixon's test assumes that, without the suspected outlier, the data are normally distributed. Therefore, it is necessary to perform a test for normality on the data without the suspected outlier before applying this test.

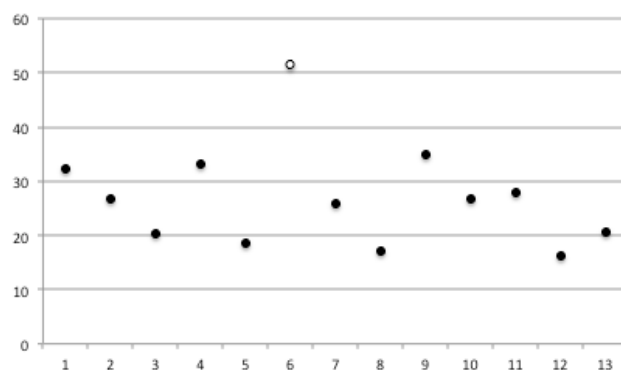
If the data are not normally distributed (see Chapter 6), it may be necessary to carry out a transformation of the data (see Chapter 7).

EPA QA/G-9 indicates that Dixon's Test can be applied when more than one outlier is present. If the test is carried with a second suspected outlier is present the test may lead to masking where two or more outliers close in value may mask their presence. Therefore, if the Extreme Value test is to be used for multiple outliers apply the test to the least extreme value first.

4.2.1.1 Visualisation of the data

Plot the data as a simple time series to enable a visual assessment to be carried out. Drawing 6.4 shows a data set that indicates that one value, the sixth sample, might be a possible outlier.

Drawing 6.4. A series thirteen measurements of which the sixth value appears to be a potential outlier.



4.2.1.2 Check that the data set without the outlier is normally distributed.

Chapter 6 of the handbook provides methods for assessing the normality of a distribution. This data set has been used as the example data in Chapter 6 and it is confirmed as being normally distributed.

4.2.1.3 Selection of the appropriate form of Dixon's equation

Dixon's test has six different equations to apply to the data. The choice of which equation depends on a combination of

- i. whether the outlier is low compared with the remainder of the data, Case 1, or high compared with the remainder of the data, Case 2.
- ii. the number of values in the data set (n), including the extreme value being evaluated

Table 4.2 shows the six equation and the conditions under which each one is to be used.

Table 4.2. Dixon's six equations used in the Extreme Value Test

n	Low Outlier	High Outlier
$3 \leq n \leq 7$	$C = \frac{(X_{(2)} - X_{(1)})}{(X_{(n)} - X_{(1)})}$	$C = \frac{(X_{(n)} - X_{(n-1)})}{(X_{(n)} - X_{(1)})}$
$8 \leq n \leq 10$	$C = \frac{(X_{(2)} - X_{(1)})}{(X_{(n-1)} - X_{(1)})}$	$C = \frac{(X_{(n)} - X_{(n-1)})}{(X_{(n)} - X_{(2)})}$
$11 \leq n \leq 13$	$C = \frac{(X_{(3)} - X_{(1)})}{(X_{(n-1)} - X_{(1)})}$	$C = \frac{(X_{(n)} - X_{(n-2)})}{(X_{(n)} - X_{(2)})}$
$14 \leq n \leq 25$	$C = \frac{(X_{(3)} - X_{(1)})}{(X_{(n-2)} - X_{(1)})}$	$C = \frac{(X_{(3)} - X_{(1)})}{(X_{(n-2)} - X_{(1)})}$

4.2.1.4 Calculation of the Test Statistic

In this example we have thirteen values and have a suspected high outlier and so the High Outlier and $11 \leq n \leq 13$ equation is the one to use.

$$C = \frac{(X_{(n)} - X_{(n-2)})}{(X_{(n)} - X_{(2)})}$$

Sort the data into ascending order produces the following series:

16.3	17.0	18.47	20.27	20.5	26.0	26.7	26.87	27.8	32.15	33.22	34.8	51.4
------	------	-------	-------	------	------	------	-------	------	-------	-------	------	------

Substituting into the equation above and solving gives -

$$C = \frac{(51.4 - 33.22)}{(51.4 - 17.0)} = \frac{18.18}{34.4} = 0.5285$$

4.2.1.5 Evaluation of the Test Statistic

Compare the calculated value of C with the test value given in Table A-3 corresponding to the number of samples, n and the specified level of confidence, α .

For values of $n=13$ and $\alpha=0.05$ the critical value = 0.521

Since the calculated C is greater than the critical value it is concluded that the value of 51.4 is an extreme value at a confidence level of 95%.

If the calculated C had been less than the tabulated value then the conclusion would have been that the value is possibly a statistical outlier at a confidence level of 5%.

4.3 Outliers and Trend Analysis

Section 10 of this Handbook introduces the detection and analysis of spatial and temporal trends. When outliers and extreme values are included in the regression analysis there is the possibility for trends to be exaggerated or reduced.

Outliers that are retained within the dataset and are a result of the underestimation of the true environmental variability should be included in trend analysis.

Outliers that are retained but have been identified as infrequent gross pollution events or extreme natural environmental events should be carefully evaluated before being included in the data set used for the trend analysis.

For pollution events it may be appropriate to carry out a trend analysis on the number of gross pollution events to determine whether the number each year is increasing.

5 Values below the Limit of Detection

5.1 Introduction

Data generated from chemical analysis may fall below the limit of detection limit (LoD) of the analytical procedure. During the preparation of the monitoring plan discussions with the laboratory will have made attempts to minimize the frequency of occurrence but in some cases there will be occasions when the environmental concentrations are below the LoD.

As discussed during Training 1 and 2 a 'rule of thumb' for specifying a particular LoD is that the LoD should be $\leq 10\%$ of the concentration of interest. This usually means $\leq 10\%$ of the environmental standard of interest. Setting such a level will ensure two important conditions are managed:

- i. Progressive increases in concentration towards the environmental standard can be tracked.
- ii. If the standard deviation of the determinand is large and the LoD is close to the environmental standard it is difficult to confirm, statistically, that the water quality complies with the standard.

Information that the concentration is below a particular concentration remains valuable information but is not quantitative. The presence of measurements below the limit of detection prevents the calculation of a mean and standard deviation unless some form of intervention is carried out.

Ignoring the less than values when calculating a mean and standard deviation will bias the estimate of the mean upwards and the estimate of the standard deviation downwards. The extent of the bias will be greater the greater the proportion of less than values in the data set.

5.2 Reporting of Values below the Limit of Detection

These measurement data are generally described as not detected (nd) or non-detects. The practice of reporting them as zero or not present (np) should be discouraged. Where np or nd is shown in a table a footnote should indicate the limit of detection for that particular determinand. The use of the less than symbol (<) followed by lod concentration may also be used. This is particularly useful when using the water quality data analysis package AARDVARK (WRc,1989) which will recognize the symbol and ask what action should be taken with regard to substitution.

In cases where measurement data are described as not detected, the concentration of the chemical is unknown. The only certainty is that it lies somewhere between zero and the detection limit.

5.3 Making use of values below the limit of detection

There are a number of ways to include the information contained in data sets that contain values indicated as being below the detection limit.

There is no single procedure that is applicable in all cases. Some general guidelines that depend on the proportion of less than values contained in the data set are presented in Table 5.1

Table 5.1. Options for management of value reported as no-detects.

Percentage of non-detects	Approach
<15%	Replace with LoD/2, DL or a small number.
15%-50%	Trimmed Mean, Cohen's adjustment, Winzorised mean and standard deviation.
>50%-90%	Calculate a percentile value.
>90%	Review the objectives of the monitoring plan

5.4 <15%: Replacement.

This process involves simply replacing the non-detect values with a teal number. The choice of whether to use zero, the LoD or LoD/2 can make small differences in the calculation of the mean and standard deviation.

- Replacement by zero will underestimate the mean and overestimate the standard deviation.
- Replacement by the LoD will overestimate the mean and underestimate the standard deviation.
- Replacement by LoD/2 will be mid-way between the two.

The most important thing is to be consistent with the replacement model that is used and to record what replacement was adopted.

Following replacement a mean and standard deviation can be carried out in the normal way.

5.5 15%-50%: Cohen's Adjustment

5.5.1 Introduction and assumptions

The Cohen's procedure for data sets containing 15%-50% non-detects is the one which will be described in this handbook since it provides both estimates of mean and variance which can be used subsequently for statistical analysis.

The method provides adjusted estimates of the sample mean and variance that takes account of data below the limit of detection. The adjusted mean and variance can then be used in the normal way for comparison with standards or between stations.

Cohen's method requires that the data without the non-detects be normally distributed and the detection limit was the same throughout the data collection period.

Directions for Cohen's method are contained in the following steps.

5.5.2 Confirmation of Normality.

Determine whether the data greater than the LoD are normally distributed (See Chapter 6). If the data are not normal, carry out a data transformation (See Chapter 7). Following transformation confirm that normality has been established.

5.5.3 Calculate initial statistics of the data set.

- Determine the total number of measurements = n
- Determine the number of measurements > LoD = m
- Calculate the mean of all the values greater than LoD = $\bar{X}_d$
- Calculate the variance of all the values greater than LoD = s_d^2

5.5.4 Determine the adjustment factor $\hat{\lambda}$

5.5.4.1 Calculation of Table A10 parameters

- Calculate $h = \frac{n-m}{n}$
- Calculate $\gamma = \frac{s_d^2}{(\bar{X}_d - LoD)^2}$

If $\gamma > 1$ then it is likely the distribution is not normal or has not responded to transformation. However there will be a tendency for γ to approach and exceed the value of 1 as the percentage of <LoD values approaches 50% even with a normally distributed data set.

Refer to table A-10 to determine the adjustment factor $\hat{\lambda}$ using h and γ to select the appropriate column and row to select the value of $\hat{\lambda}$.

5.5.4.2 Single and double interpolation

Where the values of h and γ do not coincide exactly with the column and row identifiers in the table single or double interpolation will be required.

Interpolation is a method of constructing new data points within the range of a discrete set of known data points or values. The requirement frequently occurs when using tables of look up values when the calculated input value lies between the tabulated column or row input values.

Table A-10 provides values corresponding to column and row values of h and γ at 0.05 intervals.

As an example for calculated values of h=0.20 and γ =0.10 the corresponding value of $\hat{\lambda}$ from the body of the table is 0.225741 as shown in Figure 5.1.

Figure 5.1 Extract from Table A-10 Values of the Parameter For Cohen's Adjustment.

Y	h	
	0.20	0.25
0.05	0.25033	0.32793
0.10	0.25741	0.33662

However if the calculated h and γ do not correspond to the row and column labels, such as h=0.21 and γ =0.08 interpolation is required.

Figure 5.2 shows an extract from and Microsoft Excel spreadsheet used to carry out one and double interpolation. Columns I and K contain the $\hat{\lambda}$ for values of h=0.20 and 0.25 for the values of γ either side of the calculated γ =0.08.

The equations shown in Rows 11 and 13 interpolate horizontally calculating the values corresponding to h=0.21. The equation in Row 12 interpolates vertically between the values above and below to generate a value corresponding to γ =0.08.

Figure 5.2. Equations used to carry out single and double interpolation.

	G	H	I	J	K	L
8						
9						
10		Y	0.20	0.21	0.25	
11		0.05	0.25033	=I11+(K11-I11)/(K10-I10)*(J10-I10)	0.32793	
12		0.08		=J11+(J13-J11)/(H13-H11)*(H12-H11)		
13		0.10	0.25741	=I13+(K13-I13)/(K10-I10)*(J10-I10)	0.33662	
14						

The same table is shown in Figure 5.3 but in this case the equations in Column J are replaced by the calculated values of $\hat{\lambda}$. are shown with the value for h=0.21 and γ =0.08 being 0.27029

Figure 5.3. Table A-10 showing interpolated values.

	G	H	I	J	K	L
8						
9						
10		Y	0.20	0.21	0.25	
11		0.05	0.25033	0.26585	0.32793	
12		0.08		0.27029		
13		0.10	0.25741	0.27325	0.33662	
14						

5.5.4.3 Calculation of adjusted mean and adjusted variance.

- Calculate the adjusted mean

$$\bar{X} = \bar{X}_d - \hat{\lambda}(\bar{X}_d - LoD)$$

- Calculate the adjusted variance

$$s^2 = s_d^2 + \hat{\lambda}(\bar{X}_d - LoD)^2$$

The adjusted variance is then converted to the adjusted standard deviation by taking the square root and, together with the adjusted mean can be used as normal.

5.6 >50% -90%: Calculate a percentile value.

If more than 50% the analysed samples are below the limit of detection it is not possible to calculate a meaningful mean of the data set. Instead it is necessary to calculate a percentile value to characterize the environmental concentration. This can be carried out using the Excel function PERCENTILE(data array, required percentile). The required percentile is entered as a decimal proportion, eg 90% is entered as 0.9.

Data below the LoD must be replaced by a number since the PERCENTILE function will ignore cells containing non-numeric characters and so will return an incorrect value. The replacement value must be less than the lowest measured value in the data set.

CAUTION: Attempts to calculate a percentile which is less than the percentage of less than values in the data set will return an incorrect value – in fact it will be the replacement value.

5.7 Greater than 90%:

When there are more than 90% non-detect values in the data set it may be necessary to review the objectives of the monitoring plan.

If the objective is to quantify only the maximum concentration then a single measurement above the limit of detection is sufficient to meet the objectives of the plan.

If however the objective is to determine whether the environmental concentration is below the limit of detection then 100% of samples being reported as <LoD this is sufficient to meet the objectives of the plan.

5.8 Non-detects and trend analysis

Regression models utilise paired numeric values to compute the values of the trend parameters, the slope and intercept values. Non-detects should be replaced before carrying out any trend analysis since the occurrence of a low, but unknown, concentration contributes to the trend. In this case replacement by LoD/2 is an appropriate substitution.

Since each of the replacement values are identical their presence exerts a disproportionate effect on, not only the slope and intercept but also the correlation coefficient. The greater the proportion of non-detects the greater their influence on the trend statistics.

Judgment of the effect the number of non-detects on the trend statistics on a case by case is strongly recommended.

6 Testing for Normality

6.1 Introduction

The assumption that the data are normally distributed is very important because it is the basis for the majority of statistical tests.

A normal distribution is one of the most common probability distributions encountered during the analysis of environmental data and is a reasonable model for the behavior of certain random events.

In addition, the Central Limit Theorem states that as the sample size becomes large, some of the sample summary statistics, for example the sample mean, behave as if they are a normally distributed variable. As a result, a common assumption associated with parametric tests or statistical models is that the errors associated with data conform to a normal distribution.

Tests for normality fall into two groups, graphical and numerical.

6.2 Graphical Method.

Graphical methods present information about data sets that may not be apparent from simply looking at a table of numbers or a test statistic. A histogram is probably the most useful form of presentation for determining whether or not data follow a normal curve.

Tools for constructing a histograms are available in the Excel Data Analysis Tool Pac and also in StatPus:Mac.

Using a plot to decide if the data are normally distributed involves making a subjective decision. For extremely non-normal data, it is easy to make this determination; however, in many cases the decision is not straightforward. Therefore, formal test procedures are usually necessary to test the assumption of normality.

6.3 Numerical Methods

6.3.1 Introduction

Several numerical methods are available for testing data for normality. Some can be calculated easily within Excel while others require access to more advanced statistical software.

One of the most powerful tests is the Shapiro-Wilk test for normality but this requires access to the advanced software. However the Studentized Range Test, in most cases, performs as well as the Shapiro-Wilk test and is much more easily carried out.

The features of the tests that are included in this handbook are summarized in the following Table 6.1. Both are easily calculated and can be used with small to medium data sets (upto 1,000 data points)

Table 6.1. Comparison of Coefficient of Variation and Studentized Range Test for testing for normality

Test	Sample Size	Comment
Coefficient of Variation	No limits	A test that can be used quickly to discard the assumption of normality – ie. confirm that the data are not normally distributed.
Studentized Range Test	$\leq 1,000$	Frequently as efficient as Shapiro-Wilk. Test confirms whether data are normally distributed. Does not perform well if the data are asymmetric with distinct tails to the data.

6.3.2 Coefficient of Variation

6.3.2.1 Introduction and assumptions

The coefficient of variation (CV) may be used to determine very quickly whether or not the data follow a normal curve by comparing the sample CV to 1.

If the calculated CV is greater than 1, the data should not be regarded as normally distributed. However, this method should not be used to conclude the opposite. That is, if the calculated CV is less than 1 it should not be concluded that the data are normally distributed.

This test should be used only in conjunction with other statistical tests or when the graphical representations of the data indicate extreme departures from normality.

6.3.2.2 Calculation of CV

- Calculate the sample mean $\bar{X}$ and sample standard deviation s .
- Calculate coefficient of variation $CV = \frac{s}{\bar{X}}$

If $CV > 1$ the data are not normally distributed

If $CV < 1$ It can not be concluded that the data are normally distributed. Use a graphical or another test.

6.3.3 Studentized Range Test

6.3.3.1 Introduction and assumptions

The Studentized Range Test compares the range of the sample values, the difference between the maximum and minimum values, to the sample standard deviation. Tables of critical values for sample sizes up to 1,000 are given in Table A-2 of Appendix A and are used for determining whether the absolute value of this calculated statistic is significantly large.

The studentized range test does not perform well if the data are asymmetric and if the tails of the data are heavier than the normal distribution. The test can be sensitive to extreme values. Unfortunately, positively skewed data, which are common in environmental applications, have these characteristics. If the data appear to be positively skewed then this test should not be used.

In most cases, the studentized range test performs as well as the Shapiro-Wilk test and is much easier to apply and when used in conjunction with the CV test.

6.3.3.2 Calculation of test statistic

- Calculate the sample range (w) and sample standard deviation (s).
- Calculate:
$$\frac{w}{s} = \frac{X_{(n)} - X_{(1)}}{s}$$

6.3.3.3 Evaluate the test statistic

- Obtain the critical values which are contained in Table A-2 corresponding to the Level of Significance (α) and the number of data values (n). The table provides two numbers, a and b .
- Compare the calculated statistic with the two critical values. If the calculated statistic lies between the two critical values then the assumption of normality is not rejected. That is, the data can be considered to be normally distributed.

7 Transformation to Normality

7.1 Introduction

Most statistical tests are based on assumptions about the data to which they will be applied. For example, common assumptions are that the data are normally distributed, that the two independent data sets have equal variances and that there are no underlying spatial or temporal trends in the data set.

If the data do not meet such assumptions, then the result of a statistical test may be biased or incorrect. However, in many cases, data that do not satisfy the basic assumptions can be transformed mathematically into a form that results in normality thereby allowing standard statistical tests to be carried out.

7.2 Transformation Procedure

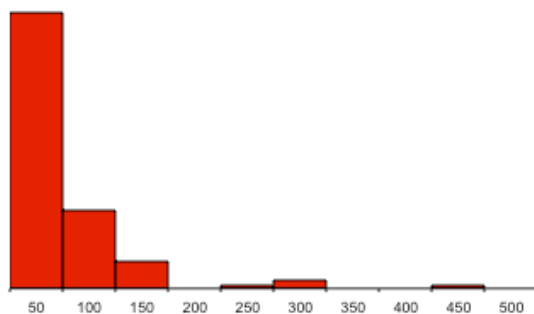
If a histogram of the data set indicate that the values do not conform to a normal distribution and/or the statistical tests for normality described in Chapter 6 indicate that the values are not normally distributed then a transformation should be carried out. Following transformation the resulting data set should be tested to confirm that normality has been established.

7.2.1 Positive Skew or Log Normal Distribution

7.2.1.1 Shape and occurrence of the distribution

The positive skew is characterized by the presence of a right hand tail of the distribution having been extended as demonstrated by the histogram shown in Drawing 7.1.

Drawing 7.1. Positive Skew Distribution showing the long tail to the right



This form of distribution is very common for data sets relating to microbiological counts, total-coliforms, faecal coliforms, *E.coli* etc.

7.2.1.2 Logarithmic Transformation

The logarithmic ($\log X$ or $\ln X$) may be used when the original measurement data follow a +ve skew or lognormal distribution. As shown in the histogram in Drawing 7.1.

The requirement for a log transformation is also indicated when the standard deviation:mean ratio is a constant. For example if the standard deviation of data around

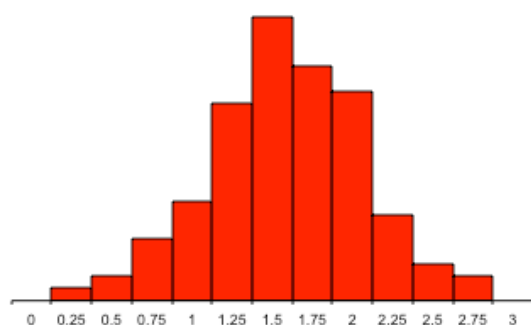
30 mg/l is approximately 3 mg/l, and the standard deviation of the data around 60mg/l is approximately 6 mg/l, then a logarithmic transformation would be required. This in contrast to a normal distribution for which the standard deviation is independent of the concentration at which it was measured.

The logarithmic base, either base 10 or natural, must be applied throughout the whole of the data analysis process. When comparing the data with an environmental standard for assessment of compliance the numerical value of the standard must also be transformed in the same way. For example if the data are $\text{Log}_{(10)}$ transformed then a standard of 5mg/l must also be transformed; $\text{Log}_{(10)}$ of 5mg/l being 0.6990.

If some of the original values are zero, as may be the case when working with microbiological counts it is necessary to replace the zero values by a small non-zero value since the logarithm of zero does not exist. The magnitude of the non-zero value depends the values of the smallest non-zero count in the data set. As a general rule, a value of one-tenth the smallest non-zero value should be used.

In the example shown in Drawing 7.1 there were no zero counts and so a $\text{Log}_{(10)}$ transformation could be applied directly to the data set with the histogram of the transformed data demonstrating a normal distribution as shown in Drawing 7.2.

Drawing 7.2. Distribution in Drawing 7.1 following Log-transformation.



The dataset following transformation now follow a classical normal distribution bell curve. Following such successful transformations the data, in its transformed state can be subjected to analysis.

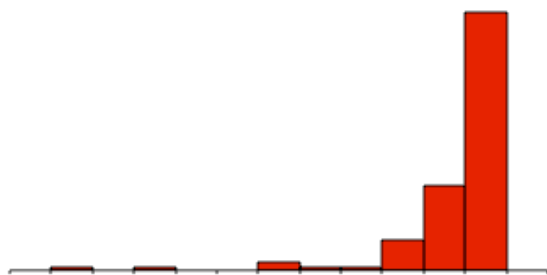
7.2.2 Negative Skew Distribution

7.2.2.1 Shape of the negatively skewed distribution

Very occasionally a data set may be encountered which shows a negative skew in which the left hand tail is drawn out. Such data set will have a histogram taking the form shown in Drawing 7.3.

Attempts to normalize such a data set directly usually prove unsuccessful. However if the data are reflected then a positive skew distribution will be created. The values in the reflected data set can then be transformed using a Log transformation.

Drawing 7.3. Typical shape of a negatively skewed distribution.



7.2.2.2 Reflection transformation.

The reflected data set is created by subtracting the individual measured values from a constant. The reflection constant is chosen to be slightly greater than the maximum value in the data set. For example if the maximum measured value is 5.3 mg/l a suitable reflection constant would be 7.0 mg/l. Choosing 7.0 rather than 6.0 will ensure that all of the values in the new data set are greater than one which will result in subsequent Log_{10} transformations generating values >1 and any square root transformations being carried out on numbers greater than one.

7.3 Post Transformation actions

Transformation and confirmation that the transformed data conform to a normal distribution will ensure the validity of F-tests, t-tests etc carried out subsequently.

No attempt should be made to transform the data back to the original form because this can lead to biased estimates. For example, estimating quantities such as means, variances and confidence limits in the transformed scale will lead to biased estimates when transformed back into original scale.

7.4 Additional reasons for carrying out transformations.

Transformations may also make the analysis of data easier by changing the scale into one that is more familiar or easier to work with. This is particularly useful when plotting a scatter diagram between two pollutants whose concentration data is positively skewed resulting in 'clumping' of data points close to the common zero origin of the two axes. When the data sets are large this can result in overlapping of data point on the scatter plot.

Reformatting of both axes from linear to log scale will in most cases enable the relationship between the two variables in the lower part of the concentration range to be resolved.

8 Testing for Compliance with a Standard

8.1 Introduction

Probably the most frequent question that a water quality monitoring plan is asked to answer is whether the water quality meets the standard applicable to the beneficial use category assigned to the water body.

The question may be asked in relation to the annual mean, the annual maximum or any individual measurement.

8.2 Comparison of an Annual Mean Concentration with the Standard.

8.2.1 Basis of the test.

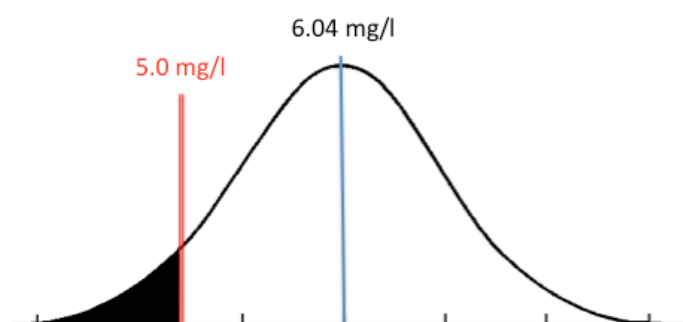
The basis of the test is to determine the probability that the calculated average, for which the variability can be calculated, exceeds a particular concentration, in this case the environmental standard. The question being asked is best demonstrated by reference to Drawing 8.1 which is based on the following scenario.

Seven samples were taken over the period of one year to determine whether the river water quality complied with the national standard appropriate to the beneficial use class of the river which in this case is 5mg/l. Previous analysis of the data for dissolved oxygen concentrations in the river showed that the data could be considered to be normally distributed.

The average concentration was calculated to be 6.04 mg/l and the standard deviation of the seven samples is 1.7mg/l.

Since the objective is to compare the annual average with the water quality standard it is necessary to use the standard error of the mean rather than the standard deviation of the individual samples. The standard error of the mean is calculated by dividing the standard deviation of the individual samples and dividing it by the square root of the number of samples.

Drawing 8.1 represents the question being asked. Given an estimated mean of 6.04mg/l, sample standard deviation of 1.7mg/l and a standard error of the mean of 0.64 what is the probability of the true mean being less than 5mg/l.



The objective is therefore to determine the probability of the true being in the black area to the left of the 5mg/l line.

8.2.2 Procedure for the test

8.2.2.1 Calculate the basic statistics for the data set.

- Calculate the annual average dissolved oxygen concentration = $\bar{X}$
- Calculate the standard deviation of the sample set = s
- Calculate the Standard Error of the Mean $SE_{\bar{x}} = \frac{s}{\sqrt{n}}$

8.2.2.2 Evaluate the probability of the true value being below the standard.

- Use the Microsoft Excel NORMDIST function to calculate the probability that a value from a distribution with a mean value of 6.04 and a standard error of the mean is less than 5mg/l.

NORMDIST calls for four input values

- i. The test value, in this case 5.0
- ii. The mean of the dataset in this case 6.04
- iii. The standard deviation, in this case the se of the mean is used, 0.64
- iv. TRUE to indicate that the cumulative probability is required.

Using the values above the NORMDIST function returns the value 0.05

This is interpreted as the probability of the true mean lying below 5.0 mg/l is 0.05 and by inference the probability that it lies above 5.0 mg/l is 0.95. The conclusion is therefore that the annual average dissolved oxygen concentration does meet the national standard at a confidence level of 95%.

8.3 Comparison of a single measured concentration with the Standard.

8.3.1 Basis for the test

The objective of this test is to determine whether single value complies with the relevant standard. The single value could be the annual maximum or each of the individual measurements taken during the year.

Single measured values provide no information about the environmental variability at the time of sampling. In a highly dynamic system such as a river variability will be different under different flow conditions, different seasons, different tidal states and in navigable river different boat traffic conditions. Ideally, replicate samples are taken at the time of sampling to provide measure of the environmental variability, or environmental standard deviation. Thereby providing a mean and standard deviation with which to carry out the statistical test.

In the absence of a sample specific standard deviation, one calculated previously from replicate samples taken under similar seasonal and hydraulic conditions, could be used but the similarity of environmental conditions can not be guaranteed.

The variability around a single measurement can be reduced by creating a composite sample on site. This would be achieved by taking multiple samples in the field. These would be combined using equal size aliquots from each sample to provide a single

sample for analysis. Although this procedure will improve the accuracy of the estimate it will still not provide a measure of the variability.

The fallback position is to use the laboratory standard deviation measured as part of their QA/QC procedure. This is a far from ideal situation since the standard deviation generated by this procedure is the standard deviation associated with taking, say 100ml, analytical sub-samples from a 2.5 litre bottle and bears little relationship to the much larger variation found in the environment.

The procedure for carrying out the comparison is exactly the same as that described in Section 8.2 above, which uses the Excel Function NORMDIST and in this case the standard deviation of the sample is used. If that is not available a similar conditions value is used and as a last resort the laboratory standard deviation.

9 Differences Between Locations or Times

9.1 Introduction

Typically this procedure would be used to determine whether a significant difference exists between the measurements taken at a pair of sampling stations upstream and downstream of an industrial or municipal outfall or upstream and down stream of a confluence of two rivers.

The location of, and the sampling design at the downstream sampling is critical in obtaining the best estimate of the downstream concentration. This is achieved by ensuring that the station is downstream of the outfall or confluence mixing zone or that samples are taken across the width of the river to create a composite sample that is representative of the average cross sectional concentration.

Comparison of samples taken at different times is carried out when comparing the samples taken in one year with the samples taken in a different year. This is used to investigate long term chronic deterioration, or improvement, in water quality or to evaluate the effect of a change in the quality of a discharge or the introduction of a new discharge.

Evaluating differences in the concentrations measured at two different stations or at two different times follow the same procedure.

The measurements are estimates of the true concentrations and as such are subject to random variability and have an associated variance. The statistical test compares the difference between the two sets of data with variances of the measurements to determine the probability that the estimates are not different

9.2 Assumptions for the comparison.

The assumptions are that the two sets of data are normally distributed and that the variances of each set of data are equal. The stages in the process are:

- Test the data sets for normality and if not normal carry out transformation on both data sets.
- Test the data sets for equality of variance; F-test.
- If the variances are equal carry out t-test for equal variances: if the variances are not equal carry out t-test for unequal variances.

The procedures for testing for normality and any subsequent transformation, have been described in Section 6 and Section 7 respectively.

9.3 Testing for equality of variances – the F-test

9.3.1 Procedure for the F-test.

The F-test is available in both the Excel Data Analysis Tool Pack and in the DataPlus:Mac application and compares the ratio of variances of the two sets of data by computing

$$F = \frac{s_1^2}{s_2^2}$$

By convention the higher variance is s_1 and the smaller variance s_2 .

9.3.1.1 Identification of the data set with the higher variance.

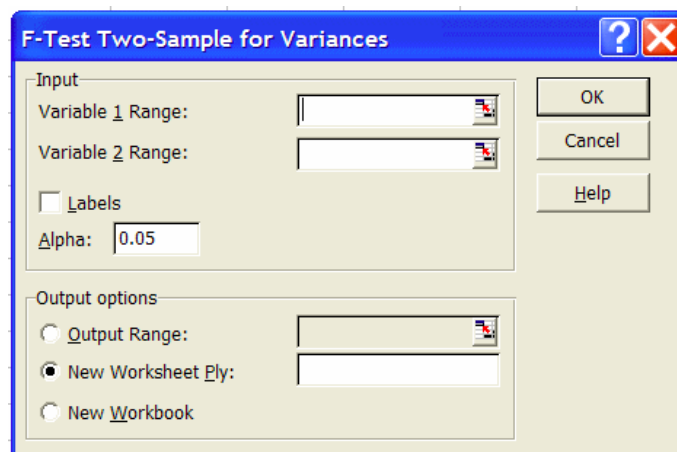
Use the Excel function =VAR(data array) to calculate the variance for each data set to determine which one has the higher variance.

Give meaningful names to each data set as a column header. This ensures that the data sets being compared.

9.3.1.2 Carry out the F-test.

Activate the F-Test Two-Sample for Variances data entry panel which is shown in Figure 9.1

Figure 9.1. F-Test Two-Sample for Variances data entry panel



Enter the data required in each of the input boxes.

- Data array with the greater variance as Variable 1
- Data array with the smaller variance as Variable 2
- Tick the Labels box
- Set the alpha value if different from the default value of 0.05
- Select the cell in the worksheet that is to be the upper left cell of the output table.
In the case of the F-test the output table is 3cx10r.

Return OK.

The resulting test output table is placed in the specified location in the worksheet and will take the form shown in Figure 9.2.

Figure 9.2. Output table for a Two Sample F-test

F-Test Two-Sample for Variances		
	Station A	Station B
Mean	6.04	8.07
Variance	2.88	0.53
Observations	7	7.00
df	6	6.00
F	5.45	
P(F<=f) one-tail	0.03	
F Critical one-tail	4.28	

The table displays the following information

- The names of the two stations confirming the data used in the analysis
- The corresponding calculated mean and variance.
- The number of observations in each data set. This will act as confirmation that the full data array has been selected for the analysis.
- The number of degrees of freedom for each data array. This is only required if the Critical F value is to be obtained from an F Distribution table (Table A-9 in EPA QA/G-9). The ToolPack generates the Critical F.
- The calculated F value, F_{calc}
- The p-value (0.03) is the probability that a value of F greater than or equal to the calculated value ($F_{\text{calc}} = 5.45$) could have occurred by chance if there were no difference in the variances. In this example the risk of getting the value $F_{\text{calc}} = 5.45$ by chance is less than $\alpha = 0.05$.
- The Critical F, $F_{\text{crit}} = 4.28$. Because $F_{\text{calc}} > F_{\text{crit}}$ ($5.45 > 4.28$), the null hypothesis is rejected at the 95% level of confidence ($100\% \times (1 - \alpha)$).

Since the variances are not equal then it is necessary to carry out the t-test for the unequal variance condition.

9.3.1.3 Carry out the t-test.

The t-test can also be called up from the Data Analysis ToolPak and presents a data input table as shown in Figure 9.3

Enter the data required in each of the input boxes.

- Data array with the greater variance as Variable 1
- Data array with the smaller variance as Variable 2
- Hypothesised Difference – this is zero since the H_0 is that there is no difference between the mean values.
- Tick the Labels box
- Set the alpha value if different from the default value of 0.05
- Select the cell in the worksheet that is to be the upper left cell of the output table. In the case of the t-test for unequal variances the output table is 3cx13r.

Return OK.

The resulting test output table is placed in the specified location in the worksheet and will take the form shown in Figure 9.4.

Figure 9.3 Data entry panel for t-Test Two-Sample Assuming Un-equal Variances

The output table shown in Figure 9.4 displays the following information

t-Test: Two-Sample Assuming Unequal Variances		
	Station A	Station B
Mean	6.04	8.07
Variance	2.88	0.53
Observations	7	7.00
Hypothesized Mean Difference	0	
df	8	
t Stat	-2.91	
P(T<=t) one-tail	0.01	
t Critical one-tail	1.86	
P(T<=t) two-tail	0.02	
t Critical two-tail	2.31	

- The names of the two stations confirming the data used in the analysis
- The corresponding calculated mean and variance.
- The number of observations in each data set. This will act as confirmation that the full data array has been selected for the analysis.
- The number of degrees of freedom used in the t-test.
- The calculated t statistic value, t_{calc}

Since the t-test does not know whether a one- or two-tail is being carried critical t values are given for both. Since the expectation is that the downstream station, Station B, will have a higher concentration a one-tail test is applicable.

- The Critical t, $t_{\text{crit}} = -1.86$. Because $t_{\text{calc}} > t_{\text{rit}}$, the null hypothesis is rejected at the 95% level of confidence ($100\% \times (1 - \alpha)$).

The conclusion is therefore that the concentration at Station B is significantly higher than the concentration at Station A at the 95% level of confidence ($100\% \times (1 - \alpha)$).

10 Testing for Trends.

10.1 Introduction

This section provides a statistical tool for recognizing trends in environmental data and subsequently quantifying them. The detection and estimation of temporal or spatial trends are frequently subsidiary questions included in many environmental monitoring plans.

10.2 Graphical Analysis

10.2.1 One-dimensional Trends

In cases where spatial or temporal patterns are strong, simple procedures such as concentration vs distance or concentration vs time plots can reveal the presence of a trend. Subsequent linear regression over distance and time can quantify the rate of change and be used to determine whether the perceived trend is statistically significant.

In more complex situations, sophisticated statistical models and procedures may be needed. For example, the detection of trends may be complicated by the interactions of long-term and short-term trends, cyclical effects (e.g., seasonal or weekly systematic variations) or step changes due to large changes in concentration over a short distance.

An initial graphical representation is recommended as the first step to identify possible trends. A plot of the data versus time is recommended for temporal data because it may reveal long-term trends and may also show other major types of trends, such as cycles or step changes.

10.2.2 Two-dimensional Trends

Where sampling stations can not be located on a one dimensional line but are located on a two dimensional plane, as in the case of groundwater sampling or coastal water sampling, a posting plot can reveal the presence of two dimensional trends.

Drawing 10.1 shows a posting plot of microbiological water quality 30 minute before high water in the vicinity of a long sea outfall. High bacterial counts are shown in the vicinity of the seaward end of the outfall. These decline towards the shore as dilution, dispersion and bacterial decay interact to reduce the concentration. The posting plot clearly shows the longitudinal and lateral spread of the plume and that the count falls to background levels before the plume reaches the shoreline.

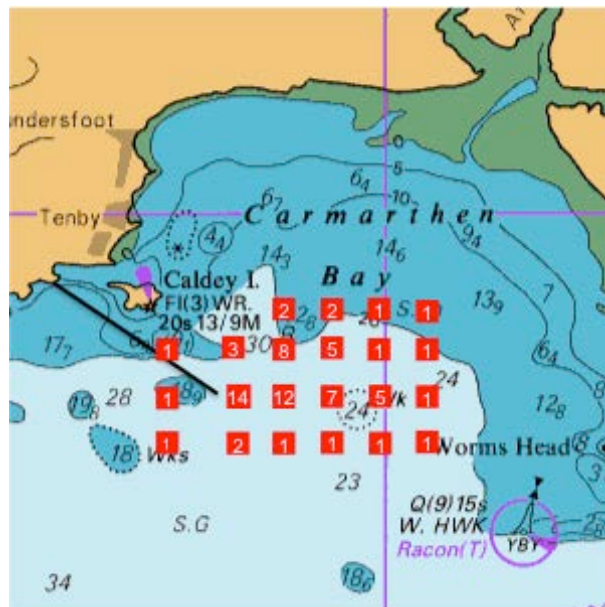
For most of the statistical approach described below the focus is on monotonic long-term trends. That is a trend which exclusively increasing or decreasing, but not both.

10.3 Estimation of a trend using the slope of a regression line

10.3.1 Application of Linear Regression

The most frequently used procedure for estimating linear trends involve regression analysis. Linear regression is a commonly used procedure in which the calculations are carried out on a data set containing paired of observations, location and concentration, or time and concentration represented by the symbols X_i and Y_i . The calculations generate the slope (a) and intercept (b) of a line that best fits the data.

Drawing 10.1. Example of a posting plot of microbiological water quality in the vicinity of a long sea outfall. (The location of the outfall and bacterial counts are for demonstration only).



For spatial trends, the X_i values represent distance along the river from a fixed upstream point and the Y_i values represent the measurement, such as contaminant concentrations. For temporal trends, the X_i values represent date and the Y_i values represent the measurement.

The regression equation takes the form $Y_i = aX_i + b$

Regression and correlation calculations are easy to apply and can be carried out in Microsoft Excel within the Chart/ScatterPlot dropdown box by invoking Insert Trendline, and Show Equation and Show Correlation.

An alternative approach is to use the three functions:

=SLOPE(Y-array, X-array)

=INTERCEPT((Y-array, X-array)

=CORREL((Y-array, X-array).

The recommended approach is to use the Chart/ScatterPlot facility since the data distribution can be seen together with the fitted trend line.

10.3.2 Limitations of Linear Regression

The application of regression models is subject to a number of limitations and assumptions.

- i. Simple linear regression is the simplest form of a mono-tonic trend. It is the most commonly used method and is designed to detect linear relationships between the two variables.
- ii. Other mono-tonic regression models include exponential and power functions.
- iii. Different forms of regression models are generally needed to detect non-linear relationships such as cyclical or non-monotonic trends.
- iv. Regression is very sensitive to extreme/outlier values and presents difficulties in handling data below the detection limit, which are commonly encountered in environmental studies. The issue of identification and the validity of extreme values has been dealt with in Section 4 of this Handbook and non-detects in Section 5.
- v. Regression also relies on two key assumptions: normally distributed errors, and constant variance, a feature of a normally distributed data set. Positively skewed data sets must be transformed before being subjected to regression analysis.

It may be difficult to verify these assumptions in practice, so the accuracy of the slope estimate may be suspect.

Because of these constraints, it is recommended that regression is not used as the only tool for estimating and detecting trends, although it can be useful as an initial, quick, and easy screening tool for identifying strong linear trends.

10.4 Significance of Linear Trends

10.4.1 Introduction

For simple linear regression, the statistical test of whether the slope is significantly different from zero is equivalent to testing if the correlation coefficient is significantly different from zero. A procedure for testing the correlation coefficient is described below.

10.4.2 Calculation of the correlation coefficient, r .

10.4.2.1 Introduction

The procedure for calculating the correlation coefficient has been described in Section 10.3.1. It is important to note that the FitTrend Line/Show correlation option provides a value of R^2 which is the Coefficient of Determination. It is necessary to take the square root of this value to obtain r , the Pearson Correlation Coefficient.

Using the function CORREL returns r which is used in the next step.

10.4.2.2 Calculate the t value

Calculate the t -value for the correlation coefficient taking into account the number of data pairs, n . This is done using the following equation.

$$t = \frac{r}{\sqrt{\frac{1-r^2}{n-2}}}$$

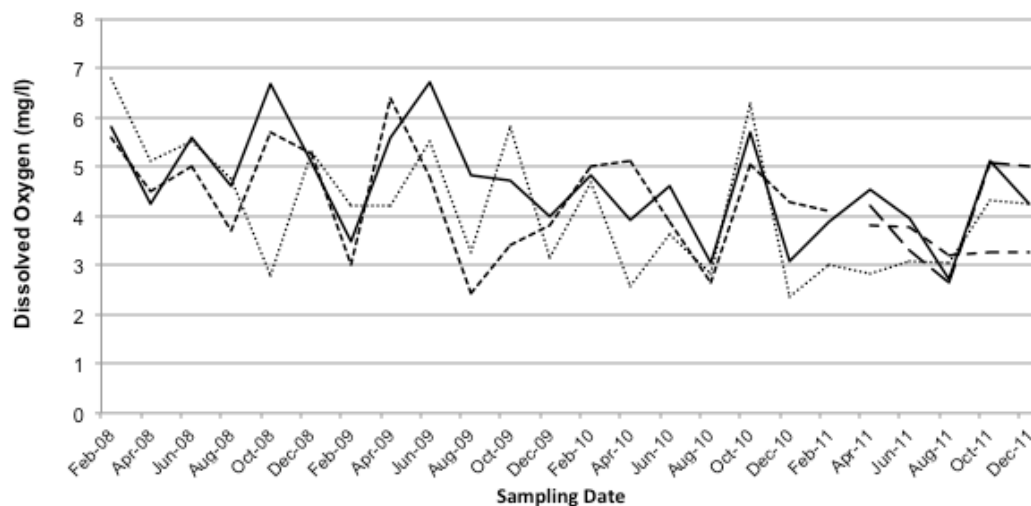
10.4.2.3 Compare the calculated t against the critical t .

The critical t is found in Table A-1. In this case the alternative hypothesis is that the r value is significantly different from zero; the direction of the difference, positive or negative is not important. Therefore the test to be carried out is a two-tailed test (see Section 3.4 of this Handbook) and at the level 95% confidence the column in the table is therefore that headed 0.975 ($1-\frac{\alpha}{2}$ where $\alpha = 0.05$). The row in the table is the one associated with $n-2$ degree of freedom.

10.5 Example of Linear Trend

Dissolved oxygen concentrations were measured over a four-year period at stations along a river passing through heavily developed urban and industrial area. The concentrations are shown plotted as a time series in Drawing 10.1 and there appears to be a downward trend in concentration over the four-year period.

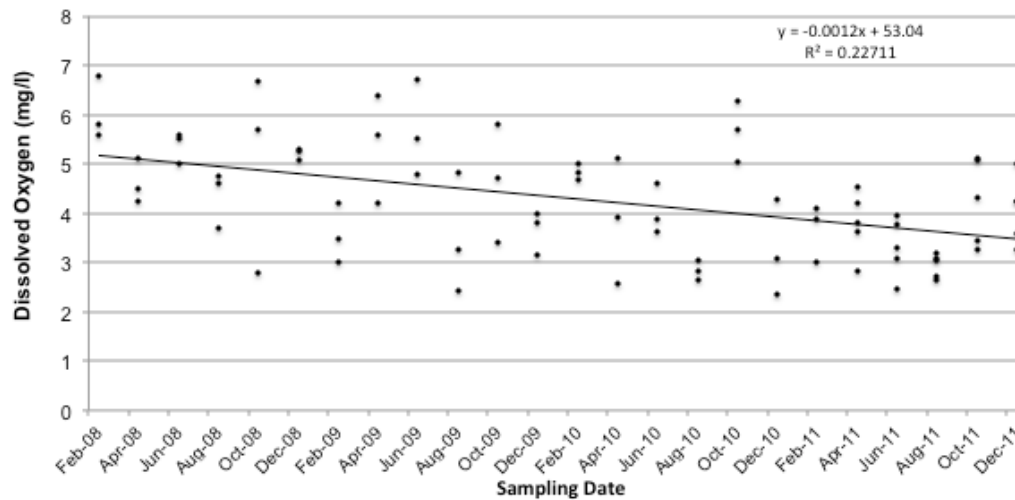
Drawing 10.1. Dissolved oxygen concentrations measured at river stations over a four year period.



For an initial examination the data from all of the stations were combined and re-plotted as shown in Drawing 10.2. A best fit linear regression model was fitted and took the form

$$Y_i = -0.0012X_i + 53.04 \text{ with an } R^2 \text{ value of } 0.227 \text{ and a corresponding } r=0.477.$$

Drawing 10.2. The data from Drawing 11.1 re-plotted as individual observations together with the best-fit linear regression line.



The high intercept value, the value of Y when X=0 is large, 53.04 mg/l. A value which is not achievable at normal atmospheric pressures and occurs because when X=0 the corresponding calendar date is 1st January 1900; the starting date of the Excel calendar. This provides a perfect example of the danger of using a linear regression to predict either forwards or backwards outside the limits of the data used to generate the equation. The linear regression model is a statistical model and not a process driven mechanistic model.

Having measured a trend in the data the next step is to determine whether the trend is significant at the 95% confidence level. As above, the equation for calculating t is:

$$t = \frac{r}{\sqrt{\frac{1-r^2}{n-2}}} = \frac{0.477}{\sqrt{\frac{1-0.227}{82-2}}} = 4.85$$

Examination of Table A-1 for $1-\alpha = 0.975$ and 80 df reveals that, by interpolation, the critical t is 1.99. Since the calculated t is greater than 1.99 the trend is statistically significant ($p=0.05$).

Appendix 1: Some Frequently Used Excel Functions

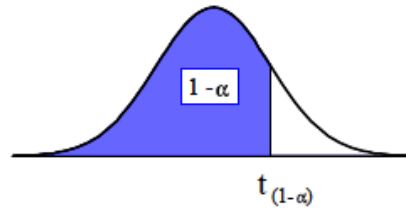
AVERAGE	Returns the average (arithmetic mean) of the arguments.
CORREL	Returns the correlation coefficient between two data sets
COUNT	Counts how many numbers are in the list of arguments
INTERCEPT	Returns the intercept of the linear regression line
MAX	Returns the maximum value in a list of arguments
MEDIAN	Returns the median of the given numbers
MIN	Returns the minimum value in a list of arguments
MODE	Returns the most common value in a data set
NORMDIST	Returns the normal cumulative distribution
PERCENTILE	Returns the k-th percentile of values in a range
SLOPE	Returns the slope of the linear regression line
STDEV	Estimates standard deviation based on a sample (ignores logical values and text in the sample)
VAR	Estimates variance based on a sample (ignores logical values and text in the sample)

Appendix 2: Tables for use in the statistical tests

The table numbers used correspond to those used in the USEPA QA/G-9 manual.

1. Table A-1: Critical Values of Student's t Distribution
2. Table A-2: Critical Values for the Studentized Range Test
3. Table A-3: Critical Values for the Extreme Value Test (Dixon's Test)
4. Table A-10: Values of the Parameter $\hat{\lambda}$ for Cohen's Estimates Adjusting for Non-detected Values

TABLE A-1: CRITICAL VALUES OF STUDENT'S t DISTRIBUTION



Degrees of Freedom	1- α								
	.70	.75	.80	.85	.90	.95	.975	.99	.995
1	0.727	1.000	1.376	1.963	3.078	6.314	12.706	31.821	63.657
2	0.617	0.816	1.061	1.386	1.886	2.920	4.303	6.965	9.925
3	0.584	0.765	0.978	1.250	1.638	2.353	3.182	4.541	5.841
4	0.569	0.741	0.941	1.190	1.533	2.132	2.776	3.747	4.604
5	0.559	0.727	0.920	1.156	1.476	2.015	2.571	3.365	4.032
6	0.553	0.718	0.906	1.134	1.440	1.943	2.447	3.143	3.707
7	0.549	0.711	0.896	1.119	1.415	1.895	2.365	2.998	3.499
8	0.546	0.706	0.889	1.108	1.397	1.860	2.306	2.896	3.355
9	0.543	0.703	0.883	1.100	1.383	1.833	2.262	2.821	3.250
10	0.542	0.700	0.879	1.093	1.372	1.812	2.228	2.764	3.169
11	0.540	0.697	0.876	1.088	1.363	1.796	2.201	2.718	3.106
12	0.539	0.695	0.873	1.083	1.356	1.782	2.179	2.681	3.055
13	0.538	0.694	0.870	1.079	1.350	1.771	2.160	2.650	3.012
14	0.537	0.692	0.868	1.076	1.345	1.761	2.145	2.624	2.977
15	0.536	0.691	0.866	1.074	1.34	1.753	2.131	2.602	2.947
16	0.535	0.690	0.865	1.071	1.337	1.746	2.120	2.583	2.921
17	0.534	0.689	0.863	1.069	1.333	1.740	2.110	2.567	2.898
18	0.534	0.688	0.862	1.067	1.330	1.734	2.101	2.552	2.878
19	0.533	0.688	0.861	1.066	1.328	1.729	2.093	2.539	2.861
20	0.533	0.687	0.860	1.064	1.325	1.725	2.086	2.528	2.845
21	0.532	0.686	0.859	1.063	1.323	1.721	2.080	2.518	2.831
22	0.532	0.686	0.858	1.061	1.321	1.717	2.074	2.508	2.819
23	0.532	0.685	0.858	1.060	1.319	1.714	2.069	2.500	2.807
24	0.531	0.685	0.857	1.059	1.318	1.711	2.064	2.492	2.797
25	0.531	0.684	0.856	1.058	1.316	1.708	2.060	2.485	2.787
26	0.531	0.684	0.856	1.058	1.315	1.706	2.056	2.479	2.779
27	0.531	0.684	0.855	1.057	1.314	1.703	2.052	2.473	2.771
28	0.530	0.683	0.855	1.056	1.313	1.701	2.048	2.467	2.763
29	0.530	0.683	0.854	1.055	1.311	1.699	2.045	2.462	2.756
30	0.530	0.683	0.854	1.055	1.310	1.697	2.042	2.457	2.750
40	0.529	0.681	0.851	1.050	1.303	1.684	2.021	2.423	2.704
60	0.527	0.679	0.848	1.046	1.296	1.671	2.000	2.390	2.660
120	0.526	0.677	0.845	1.041	1.289	1.658	1.980	2.358	2.617
∞	0.524	0.674	0.842	1.036	1.282	1.645	1.960	2.326	2.576

Note: The last row of the table (∞ degrees of freedom) gives the critical values for a standard normal distribution (z), e.g., $t = z = 1.645$.

TABLE A-2: CRITICAL VALUES FOR THE STUDENTIZED RANGE TEST

<i>n</i>	Level of Significance α					
	<i>0.01</i>		<i>0.05</i>		<i>0.1</i>	
	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>
3	1.737	2.000	1.758	1.999	1.782	1.997
4	1.87	2.445	1.98	2.429	2.04	2.409
5	2.02	2.803	2.15	2.753	2.22	2.712
6	2.15	3.095	2.28	3.012	2.37	2.949
7	2.26	3.338	2.40	3.222	2.49	3.143
8	2.35	3.543	2.50	3.399	2.59	3.308
9	2.44	3.720	2.59	3.552	2.68	3.449
10	2.51	3.875	2.67	3.685	2.76	3.57
11	2.58	4.012	2.74	3.80	2.84	3.68
12	2.64	4.134	2.80	3.91	2.90	3.78
13	2.70	4.244	2.86	4.00	2.96	3.87
14	2.75	4.34	2.92	4.09	3.02	3.95
15	2.80	4.44	2.97	4.17	3.07	4.02
16	2.84	4.52	3.01	4.24	3.12	4.09
17	2.88	4.60	3.06	4.31	3.17	4.15
18	2.92	4.67	3.10	4.37	3.21	4.21
19	2.96	4.74	3.14	4.43	3.25	4.27
20	2.99	4.80	3.18	4.49	3.29	4.32
25	3.15	5.06	3.34	4.71	3.45	4.53
30	3.27	5.26	3.47	4.89	3.59	4.70
35	3.38	5.42	3.58	5.04	3.70	4.84
40	3.47	5.56	3.67	5.16	3.79	4.96
45	3.55	5.67	3.75	5.26	3.88	5.06
50	3.62	5.77	3.83	5.35	3.95	5.14
55	3.69	5.86	3.90	5.43	4.02	5.22
60	3.75	5.94	3.96	5.51	4.08	5.29
65	3.80	6.01	4.01	5.57	4.14	5.35
70	3.85	6.07	4.06	5.63	4.19	5.41
75	3.90	6.13	4.11	5.68	4.24	5.46
80	3.94	6.18	4.16	5.73	4.28	5.51
85	3.99	6.23	4.20	5.78	4.33	5.56
90	4.02	6.27	4.24	5.82	4.36	5.60
95	4.06	6.32	4.27	5.86	4.40	5.64
100	4.10	6.36	4.31	5.90	4.44	5.68
150	4.38	6.64	4.59	6.18	4.72	5.96
200	4.59	6.84	4.78	6.39	4.90	6.15
500	5.13	7.42	5.47	6.94	5.49	6.72
1000	5.57	7.80	5.79	7.33	5.92	7.11

TABLE A-3: CRITICAL VALUES FOR THE EXREME VALUE TEST
DIXON'S TEST

n	Level of Significance α		
	0.10	0.05	0.01
3	0.886	0.941	0.988
4	0.679	0.765	0.889
5	0.557	0.642	0.780
6	0.482	0.560	0.698
7	0.434	0.507	0.637
8	0.479	0.554	0.683
9	0.441	0.512	0.635
10	0.409	0.477	0.597
11	0.517	0.576	0.679
12	0.490	0.546	0.642
13	0.467	0.521	0.615
14	0.492	0.546	0.641
15	0.472	0.525	0.616
16	0.454	0.507	0.595
17	0.438	0.490	0.577
18	0.424	0.475	0.561
19	0.412	0.462	0.547
20	0.401	0.450	0.535
21	0.391	0.440	0.524
22	0.382	0.430	0.514
23	0.374	0.421	0.505
24	0.367	0.413	0.497
25	0.360	0.406	0.489

TABLE A-10: VALUES OF THE PARAMETER FOR COHEN'S ESTIMATES ADJUSTING FOR NONDETECTED VALUES

γ	h											
	.01	.02	.03	.04	.05	.06	.07	.08	.09	.10	.15	.20
.00	.010100	.020400	.030902	.041583	.052507	.063625	.074953	.08649	.09824	.11020	.17342	.24268
.05	.010551	.021294	.032225	.043350	.054670	.066159	.077909	.08983	.10197	.11431	.17925	.25033
.10	.010950	.022082	.033398	.044902	.056596	.068483	.080563	.09285	.10534	.11804	.18479	.25741
.15	.011310	.022798	.034466	.046318	.058356	.070586	.083009	.09563	.10845	.12148	.18985	.26405
.20	.011642	.023459	.035453	.047829	.059990	.072539	.085280	.09822	.11135	.12469	.19460	.27031
.25	.011952	.024076	.036377	.048858	.061522	.074372	.087413	.10065	.11408	.12772	.19910	.27626
.30	.012243	.024658	.037249	.050018	.062969	.076106	.089433	.10295	.11667	.13059	.20338	.28193
.35	.012520	.025211	.038077	.051120	.064345	.077736	.091355	.10515	.11914	.13333	.20747	.28737
.40	.012784	.025738	.038866	.052173	.065660	.079332	.093193	.10725	.12150	.13595	.21129	.29250
.45	.013036	.026243	.039624	.053182	.066921	.080845	.094958	.10926	.12377	.13847	.21517	.29765
.50	.013279	.026728	.040352	.054153	.068135	.082301	.096657	.11121	.12595	.14090	.21882	.30253
.55	.013513	.027196	.041054	.055089	.069306	.083708	.098298	.11208	.12806	.14325	.22225	.30725
.60	.013739	.027849	.041733	.055995	.070439	.085068	.099887	.11490	.13011	.14552	.22578	.31184
.65	.013958	.028087	.042391	.056874	.071538	.086388	.10143	.11666	.13209	.14773	.22910	.31630
.70	.014171	.028513	.043030	.057726	.072505	.087670	.10292	.11837	.13402	.14987	.23234	.32065
.75	.014378	.029927	.043652	.058556	.073643	.088917	.10438	.12004	.13590	.15196	.23550	.32489
.80	.014579	.029330	.044258	.059364	.074655	.090133	.10580	.12167	.13775	.15400	.23858	.32903
.85	.014773	.029723	.044848	.060153	.075642	.091319	.10719	.12225	.13952	.15599	.24158	.33307
.90	.014967	.030107	.045425	.060923	.075606	.092477	.10854	.12480	.14126	.15793	.24452	.33703
.95	.015154	.030483	.045989	.061676	.077549	.093611	.10987	.12632	.14297	.15983	.24740	.34091
1.00	.015338	.030850	.046540	.062413	.078471	.094720	.11116	.12780	.14465	.16170	.25022	.34471

TABLE A-10: VALUES OF THE PARAMETER FOR COHEN'S ESTIMATES ADJUSTING FOR NONDETECTED VALUES

γ	h											
	.25	.30	.35	.40	.45	.50	.55	.60	.65	.70	.80	.90
.00	.31862	.4021	.4941	.5961	.7096	.8388	.9808	1.145	1.336	1.561	2.176	3.283
.05	.32793	.4130	.5066	.6101	.7252	.8540	.9994	1.166	1.358	1.585	2.203	3.314
.10	.33662	.4233	.5184	.6234	.7400	.8703	1.017	1.185	1.379	1.608	2.229	3.345
.15	.34480	.4330	.5296	.6361	.7542	.8860	1.035	1.204	1.400	1.630	2.255	3.376
.20	.35255	.4422	.5403	.6483	.7673	.9012	1.051	1.222	1.419	1.651	2.280	3.405
.25	.35993	.4510	.5506	.6600	.7810	.9158	1.067	1.240	1.439	1.672	2.305	3.435
.30	.36700	.4595	.5604	.6713	.7937	.9300	1.083	1.257	1.457	1.693	2.329	3.464
.35	.37379	.4676	.5699	.6821	.8060	.9437	1.098	1.274	1.475	1.713	2.353	3.492
.40	.38033	.4735	.5791	.6927	.8179	.9570	1.113	1.290	1.494	1.732	2.376	3.520
.45	.38665	.4831	.5880	.7029	.8295	.9700	1.127	1.306	1.511	1.751	2.399	3.547
.50	.39276	.4904	.5967	.7129	.8408	.9826	1.141	1.321	1.528	1.770	2.421	3.575
.55	.39679	.4976	.6061	.7225	.8517	.9950	1.155	1.337	1.545	1.788	2.443	3.601
.60	.40447	.5045	.6133	.7320	.8625	1.007	1.169	1.351	1.561	1.806	2.465	3.628
.65	.41008	.5114	.6213	.7412	.8729	1.019	1.182	1.368	1.577	1.824	2.486	3.654
.70	.41555	.5180	.6291	.7502	.8832	1.030	1.195	1.380	1.593	1.841	2.507	3.679
.75	.42090	.5245	.6367	.7590	.8932	1.042	1.207	1.394	1.608	1.851	2.528	3.705
.80	.42612	.5308	.6441	.7676	.9031	1.053	1.220	1.408	1.624	1.875	2.548	3.730
.85	.43122	.5370	.6515	.7781	.9127	1.064	1.232	1.422	1.639	1.892	2.568	3.754
.90	.43622	.5430	.6586	.7844	.9222	1.074	1.244	1.435	1.653	1.908	2.588	3.779
.95	.44112	.5490	.6656	.7925	.9314	1.085	1.255	1.448	1.668	1.924	2.607	3.803
1.00	.44592	.5548	.6724	.8005	.9406	1.095	1.287	1.461	1.882	1.940	2.626	3.827

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APPENDIX-4

GUIDELINED FOR WRITING ENVIRONMENTAL MONITORING REPORTS

SCOWEM

Project for Strengthening Capacity of Water Environmental Management in Vietnam

Output 2-1: Water Quality Monitoring

Guidelines for Writing Environmental Monitoring Reports

November 2012

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Appendix A Example structures for factual, instructive and leading reports.

Abbreviations

Abbreviation	Meaning
A3	ISO Paper size (420 mm x 297 mm)
A4	ISO Paper size (297mm x 210 mm)
AA	Atomic Absorption
BOD	Biological Oxygen Demand
DO	Dissolved Oxygen
<i>E coli</i>	<i>Escherischia coli</i>
EA	Environment Agency
GIS	Geographic Information System
GQA	General Quality Assessment
NGO	Non Governmental Organisation
QA	Quality Assurance
QM	Quality Management
RAF	Royal Air Force
ToC	Table of Contents
TSS	Total Suspended Solids
WQ	Water Quality
WQI	Water Quality Index
WQO	Water Quality Objective
WQS	Water Quality Standard

Summary

These guidelines have been written as one part of a project aimed at developing the capacity of staff in the five DONRE who are carrying out environmental monitoring. Although the DONRE have responsibilities for the whole of the environment the project has targeted specifically water quality monitoring. Previous development training has addressed preparation of monitoring plans and analysis and processing of data. Reporting of the monitoring is therefore the natural conclusion to theme.

The purpose of the guidelines, and the associated training, is to introduce the concept of effective preparation of effective reports. The guidelines focus on the preparation of a report that meets the requirements of the reader, the planning of the structure of the report and the effective use of text, tables and figures. Combined, these three concepts will ensure that the report transfers the information to the reader in the most effective way.

The guidelines are divided in four principal parts. Firstly, identification of the purpose of the report and the requirements of the reader. Secondly, the materials and structure of the report. Thirdly, style and presentation. Lastly, the finalization of the report.

The guidelines are intended for both the experienced report writer to enable them to rethink their approach to the report and for the newcomer to report writing to help them over the hurdle of putting the first few words on that blank sheet of paper, or screen, that will eventually lead to their first monitoring report.

1 Introduction

These guidelines are not guidelines to writing good Vietnamese spelling and grammar. They are guidelines to thinking about and structuring a report and how to use text, tables figures etc to present information in a way that is meaningful to the reader in an un-ambiguous and easily assimilable way.

Googling the phrase 'Report Writing' or 'Writing Reports' will generate numerous web-sites providing guidance on how to write a report. Much of the guidance has been produced by universities guiding the student on how to prepare project reports in a standard format for comparison and marking purposes. Others aim to produce documents that are highly visually attractive to the reader with a view to selling a product. Some of these that relate to the mechanics of the process have been included in the Bibliography at the end of this document.

The different approaches demonstrate the fact that reports are produced for many different purposes. However they do agree on the need for a structured approach to preparation of the report.

This guide describes an approach that will result in a focused, well structured and readable document. The production of such a report relies on the consideration of three factors that are common to all types of report. These are, consideration of

- i. the purpose and the reader,
- ii. the material and structure, and
- iii. the style and presentation

In the following sections of this document the three factors will be explored in relation to the types of report that may be generated when carrying out environmental monitoring.

At all times it is important to remember that reporting is an integral part of the monitoring process. It is the conversion of days of work in the field and laboratory converting data into useful information and so is equal value to all the other components.

2 The purpose and the reader

Always allow plenty of time for the identification of purpose of the report and the audience for whom the report is being written. Once these are clearly established it will save hours of work writing and re-writing the main text later on.

2.1 Defining the purpose

Clear identification of the purpose of the report or why the report is being written will determine the type and structure of the report. There are three basic types of report.

- a) Factual
- b) Instructional
- c) Leading

2.1.1 Factual report

This is designed to provide information and is a statement of facts to give the reader an accurate record of events that have occurred or measurements that have been made. In this case summarisation of data, the calculation of basic statistics and whether environmental standards were exceeded are facts.

Reports in the factual category would therefore include.

- a) Results of collection and laboratory analysis of field samples.
- b) Statistical analysis of results to indicate compliance with standards or variation over time or space.
- c) Results of an investigation into a pollution incident.

2.1.2 Instructional report

These explain what is to be done when some change is introduced to an existing way of carrying out a task. This would include.

- a) A new method of taking river water samples.
- b) A new laboratory analytical procedure.
- c) The content of a new monitoring plan or program.

An instructional report would be a step-by-step description to inform staff about the new procedure and in the case of a field, laboratory or office SoP (Standard Operating Procedure) will follow a precise structure and be subject to strict QM (Quality Management) procedures.

2.1.3 Leading report

This is a report that is used to persuade the reader to make a decision with regard to taking an action that is being proposed. This would include.

- a) A request for an increased budget to expand the monitoring plan.
- b) A proposal for the more widespread implementation of controls on effluent discharges that are causing the river water quality to fail to meet the water quality standard.

A leading report is often the most difficult to write since the report will need to inform in order to explain, and inform and explain in order to persuade.

Once the major objective of the report has been defined any subsidiary objectives that might have been identified can be accommodated within the structure.

2.2 Identifying the reader

Who is the reader? What is known about them? Unless the report is intended for internal use, little may be known about the reader.

For a factual report it is likely to be a group of people with a wide range of background knowledge and ability. An instructional report would most likely be for co-workers with a scientific or technical training. In the case of a leading report

it may be a particular individual or a committee of scientific, technical and financial experts.

Three questions will help to define the reader.

2.2.1 What does the reader know?

Two common mistakes that are made when writing reports are:

- a) To overestimate the knowledge of the reader and thereby make the report unintelligible, or
- b) To underestimate their knowledge and not hold the reader's interest.

It is therefore important to discover how much the reader already knows about the subject of the report and to write at that level of knowledge.

2.2.2 What are the reader's attitudes?

This is particularly important when writing a leading report. As an example, a report proposing an increase in the annual budget for the monitoring plan written to the finance branch in a time of financial expansion would need to be written in a different way from one written at times of financial cut-backs.

2.2.3 What does the reader want from the report?

The target reader will have expectations of the report. These may be

- a) to be provided with raw data for their own use,
- b) to be aware of the environmental status of a particular river,
- c) to be given information on which they will make decisions,
- d) to know what monitoring work is being carried out,
- e) to be given step-by-step instructions on how to carry out a task,
- f) to be persuaded by logically written scientific argument that it is necessary to invest in a piece of new laboratory equipment.

It is only when the purpose of the report is defined and clearly understood that it is possible to start the process of planning the report.

Sometimes, it may be difficult to answer these questions, especially when the circulation list contains readers with a range of different requirements. In such cases, the report should be written for the most important reader or group of readers. It may even be necessary to consider producing more than one version of the report. With a wide distribution list containing readers with a wide range of interests it is possible that some of the readers will be content to read only the executive summary. Hence the importance of the importance of the executive summary – it is to be written with this possibility in mind.

2.3 Setting the objective

Having matched the purpose of the report to the readers, it is now possible to start to consider the detail of the content of the report.

3 Materials and structure

The report writer may have spent many days in the field collecting samples, even more days in the laboratory analyzing the samples and several weeks processing the data and writing the report.

On the other hand the reader may have only a few hours, or even less, in which to read the report. Therefore the report must present the reader with easily digested information, and only enough information to help the reader reach a decision. So the content of the report, and its structure, must be very carefully planned.

3.1 Selecting our material

The three rules to follow when deciding what to put into a report are:

- a) Keep it simple. Reject the irrelevant material, discuss the doubtful, and ensure that the essential is included.
- b) Justify any conclusions with facts, and if third party information is used in the report state their sources in the references section.
- c) Build the facts into a logical and consistent case, and by doing so the reader should arrive at the same conclusion.

3.2 Planning the structure

3.2.1 *The need for planning*

During the development of the monitoring plan the requirement for preparing and issuing reports should have been considered when considering the scheduling of activities the availability of human resources. The time for data collection, processing and reporting will have been built into the timetable to enable the issues deadlines to be met.

Time spent planning of the structure and content of the report will save time later by eliminating the need to move blocks of text between different sections of the report and reducing repetition. Structured planning with intermediate milestones, draft text stages, will ensure that the report is issued on time and of a high quality.

If the report is to be written by a team it is important that all the members of the team are involved in the planning process. This will ensure that any constraints on the production of the report are identified at the earliest possible stage in the planning process.

3.2.2 *Approach to planning*

A whiteboard or a large sheet of paper, A3 landscape, provides a convenient medium for planning.

An example of a planning sheet for a single river annual water quality monitoring report is shown in Figure 3.1

Step 1: Divide the page into columns representing the major sections of the report. The headings to these columns will probably become the headings in the report. Executive Summary, Introduction, Methodology etc.....

Step 2: Make a list under each heading of all the points to be mentioned in that section. Flag information, in particular that from outside organisations, that will be needed. Put down all ideas - less important ones will be weeded out later in the planning.

Step 3: Mark the most important points and the essential points to be put forward in the reasoning when preparing a leading report.

Step 4: Mark the least important ones, points that might be irrelevant or dilute the message or reasoning and these will probably be rejected.

Step 5: The points that remain, the unmarked ones, may be of interest to some readers. Some of these could be included in the appendices while others are rejected.

Step 6: Arrange the points in a final, logical sequence that will best meet the objective of the report. It is at this point that using a white board has an advantage over paper. Writing the points on 'post-it' notes allows them to be rearranged easily within or between columns until the structure is right.

Step 7: Having agreed the structure and content of the report, a template document should be issued to all of the authors to be used for preparing the document.

Example structures for a factual report, instructional report and a leading report can be found in Appendix A. These are indicative structures to indicate how the content may be organized into a systematic order that will aid both the writer and the reader.

3.3 Rules and guidelines

The following guidelines relate to the conventional structure of a report. Some organizations have their own 'House style' in which case the writer is constrained to follow that structure.

When writing a series of reports such as annual water quality reports and having developed a style it is advisable to continue to use that style. It is easier for the writer to follow the style and easier for the reader who can find his/her way round the report.

3.3.1 Title page

This is not necessarily the cover page. However. It is the first page inside the report and normally carries the main information about the report and should include:

- a) Title
- b) Sub-title (if any),
- c) Date,

The title page may also include

- d) Author's name and position,
- e) Distribution list.
- f) Reference number (and revision number if any)
- g) Classification (eg, confidential).

Annual river water quality report - River Blackwater 2012 -

<u>Exec Summary</u>	<u>Introduction</u>	<u>Background</u>	<u>Methods</u>	<u>Results and discussion</u>	<u>Conclusions and recommendations</u>	<u>Appendices</u>
1-Continuing improvement in W Q 2-New W QI values - river g coarse 3-Only compl assoc with rainfall. caused by construction site run off.	Changes to circulation list - Env NGO now included. Purpose of the report.. Period covered First year W QI being calculated. * Put W QI method in Appendix, Mention high rainfall in April as factor for high SS	<u>Brief summary</u> of previous years compliance and trends in change - ? Use text from exec summaries for 2005-2011 * Unusually wet spring - wettest for 23 years!!	Map of stations - use 2011 - GIS team to add new station RB5** - keep old RB06 station for ref in R+D section. Table in appendix * Field DO probe replacing Winkler - put comparison in Appendix. * AA for heavy metals from June onwards * Ref to analytical methods in appendix. <u>** get photo for appendix table</u>	Put weekly data in appendix, only include max/min/mean table in text with W QS and % compliance. Show hourly rainfall plot for 20-24 April* Show W QI values using EA scheme of coloured dots on a map. I include table of target W QI values for different W QO. I include time series plots showing annual average with max and min bars for DO BOD COD amm TSS and E coli Get data from RAP station Met officer	* Compliance continuing to improve. W QI shows river in good condition for coarse fishery. <u>* Only non compliances were below storm drain outlet on 23 April.</u> * Keep RB5 for next year program - no need to bring back RB06. ** Recommend to EA more strict controls on run off from construction sites - no silt traps on new housing development on Church St.	> W QI method > Table of stations > DO vs Winkler > Analytical methods with QA data > Report and photos of run off from Church st site
John 1 st draft by 24 th	Penny First draft by 18 th	Penny First draft by 18 th	John -Field Mike -Lab 1 st draft by 18 th			John/Mike/ Penny 1 st draft by 26 th

Figure 3.1 Example Report Planning Sheet

If required by the Quality Management system it may be necessary to include a block containing the individual document number, the revision number, together with the names and signatures of the author, the reviewer, and the member of staff authorized to issue the document.

It is important not to overcrowd the page. A clear, simple layout is always the best, keep the external cover for graphics and photographs.

3.3.2 Summary

A summary is essential if the report is long. It is NOT a re-statement of the conclusions.

It must enable readers who do not need to know the details of the report to understand:

- a) Why the report has been written
- b) The purpose of the report
- c) How the information was obtained
- d) The key facts
- e) Any conclusions and/or recommendations

For a short report a summary of less than a page may be sufficient. The summary should never be more than ten percent of the main report.

3.3.3 Table of Contents

The table of contents (ToC) of short reports may be shown on the title page. It may not even be required at all. Most reports will however require a ToC to enable the reader to find their way to sections of the report that are of particular interest to them. In such a case the ToC should always be on a separate page or pages.

A typical ToC will list the major sections or chapters, sub-sections, and appendices and give their page numbers. It should be laid out clearly so as to show the relationship between them.

Consideration should be given to the lowest level of sub-section that is shown in the ToC. In some cases Level 1 may be sufficient, while in others it may be necessary to go to Level 3. The level used will be dictated by the way that levels have been used in the structure of the main body of the report

If a summary, table of abbreviations, references and bibliography are included in the report these should be listed in the ToC in the order in which they occur in the report.

MS Word can generate the table of contents automatically by selecting the **Insert/Index and Tables** drop down menu. Once set up, the ToC can be refreshed to take account of new sections that have been added and any changes in the page numbering that results from adding new text.

3.3.4 Introduction

The introduction provides the reason for the report, and shows why it was necessary. It states what the report aims to do report, its intended readership, and the scope of the report. The shorter it is, the better.

3.3.5 Body of the report

This contains your detailed facts and findings, shows how they were arrived at, and indicates the inferences to be drawn from them, all in accordance with the plan.

The body of the report would normally contain

Background: Explain briefly the previous water quality in the river and whether it has been improving or deteriorating. Summarise the main causes of pollution and how water quality is affecting the recognized beneficial river water uses.

Methods and Materials: Explain what monitoring was carried out, how it was done, at what locations, how often was it done and what quality control (QC) procedures were in place and what methods were used to analyse the data. Detailed information regarding methodologies should be put into an appendix, unless the report is an instructional report for a particular methodology.

Results and Discussion: Detailed tables of numbers should not be included in the body of the report. It is more appropriate for these to be placed in an appendix and summary tables for figures to be used in the main body of the text. For ease of reading it is preferable to divide the results and discussion into subsections with the first level of division at the geographical level, ie, by river, lake, coastal region or aquifer. Within each geographic the division would be by determinand. Further divisions would be applied as appropriate. The objective being to enable the reader to navigate their way through the report with ease and to find their way back to a topic with the minimum of searching.

In order to avoid un-necessary repetition of figures the discussion of the compliance of each determinand with the applicable standard is best covered in the data section as the description of the determinand.

3.3.6 Conclusions

The conclusion section provides the opportunity to pull together the main points of the discussion and formalize the conclusions that can be drawn. These may be in relation to the monitoring methodology and the results of the monitoring. The conclusions must be based on facts that have been presented in the previous sections of the report.

3.3.7 Recommendations

If the purpose of the report is purely to provide facts then this section is not obligatory.

Recommendations are more likely to be followed if the benefits of implementing them are clearly set out and supported by facts presented by the monitoring plan.

3.3.8 Appendices

Some reports require detailed supporting information, or perhaps information that only some of the readers will wish to access. This information would be put into the appendices.

Typical information that would be put into appendices include:

- a) Details of attendees at meetings.
- b) Blank and, in some cases, completed questionnaire and survey sheets.
- c) Detailed financial information.
- d) Large tables that are too complex to include in the main body of the report.
- e) Supporting information
- f) Photographs
- g) Details of laboratory analytical procedures (unless the subject of the report)
- h) Figures and drawings that need to be a larger page size than the main volume to be legible.
- i) Timetables and itineraries.
- j) Project management tables.

Appendices may be bound into the main report or if particularly large bound as a separate volume.

Appendices should be listed in the table of contents showing their number and title. The contents of each appendix should be listed at the front of each appendix.

3.3.9 References and/or Bibliography

The reference list contains the details of books and articles that were consulted during the preparation of the report. The details of the source must be sufficient for the reader to be able to gain access to a copy of the document in order to verify the facts that have been used in the report.

The bibliography contains details of suggested books and articles for further reading. The documents listed are there for the reader to extend their knowledge of the subject of the report further or to provide additional background information but not information that is used in the drawing of conclusions or the formulation of recommendations.

3.3.10 Glossary or Nomenclature

Usually this section would not be included in day to day reporting of monitoring activities. If necessary is it used to describe particular technical terms that are used in a report that are too long to describe in the main body of the report or the description would disturb the information flow or argument .

4 Style and presentation

Having planned planning the report, the writing of the report is guided by the structure of the report defined by the section headings. There are two elements to the writing, style and presentation.

In MS Word the term style refers to the font, the font size, line and paragraph spacing etc. Here the term style relates to the way in which the information is presented to the reader.

The style in which the report is written will to a large extent be driven by the type of report being written.

A factual report may contain very little written text and consist largely of tables of data and graphical representation of the data.

An informative report is likely to be written using technical terminology using simple sentences.

A leading report will, by its very nature, contain large passages of written text using the language to persuade the reader through strength of argument to come to a particular conclusion.

4.1 Style

It is important to produce the first draft without evaluating every sentence as it is written. Examining each phrase and sentence as it is written will suppress the natural expression of the ideas. Once there is something on paper it is easier to correct the shape to the final document.

4.1.1 First Draft

Write the first draft for yourself. Keep within the structure for the report and write.

4.1.2 First to Second Draft

Edit the first draft, trying to read it as though you are the reader for whom the report is intended. This stage provides the opportunity to:

- a) Clear up any obvious ambiguities,
- b) Substitute short, simple words where appropriate,
- c) Choose words with which the reader will be familiar. Technical terms are a useful when writing for fellow specialists, but may confuse anyone else.
- d) Use active, rather than passive verbs, eg, 'Heavy rainfall caused increases in river flow' rather than 'Increases in river flow resulted from heavy rainfall'.

In the scientific and technical field there is a tendency to write technical and scientific reports in the passive tense rather than the active tense. The exception to this is when writing instructional reports that by their very nature are active in their purpose. Where possible the use of the active tense is more likely to give rise to action.

4.2 Presentation of Data

4.2.1 *Figures, Graphs and Tables.*

Figures and graphs are valuable tools when presenting information. Complex results can be presented in a more assimilable form and can represent many pages of numbers in a clear and succinct way. However it is important that they are used carefully.

Most technical reports use figures and tables for the presentation of data, the form and quality of the figures and tables are important in determining the readability of the report.

Only use figures and tables that add value to the report. Present the data as simply and straightforwardly as possible. Present data in the text, in a figure or in a table. Never in more than one way. It is acceptable to include detailed data in a table in an appendix and in a summary figure in the main body of the text.

Before beginning to write the report, identify the data to include. Even the most carefully prepared monitoring programs generate more data than are needed for the report. Use only data that are directly relevant to the report.

Once the data to be included have been selected decide how they can best be presented; tabulated or plotted? This is determined by the needs of the reader. If it is necessary to know exact values, tabulate the results. If relative trends are more important, use graphs.

4.2.2 *Figures*

Figures used in technical reports fall into three types, graphs, drawings, and photographs. Figures are numbered with Arabic numerals in the order of their mention, unless the mention is clearly incidental.

In the final layout they are either inserted in the text near, and preferably following, their first mention or grouped together at the back.

Prepare figures taking into account their appearance in the final printed document. Clearly the size of the printed figure including the legend cannot exceed the dimensions of the report image area. Within these limits various sizes, proportions, and arrangements of figures and text are possible. A large, complex figure may be printed on facing pages or, if an A4 size document an A3 tri-fold may be used. If there are many large figures associated with the report they can be presented as an appendix in a second volume with a different page size.

All figures must have legends. If a figure has parts (a), (b), (c), etc., it must have corresponding sub-legends. Use similar wording in the legends of related figures.

Conditions applying to a figure are normally stated as part of the legend. But when the same conditions apply, for example, to every photograph in a report, it is more appropriate to state it once in the text.

4.2.3 Graphs

Graphs should be clear and simple and comply with the following guidelines:

- a) Use few data curves as possible. It is usually best to have no more than six types of lines or data points on a graph; four is better if it can be achieved.
- b) Avoid interweaving or unrelated curves.
- c) As few words as possible should be inserted directly on the figure.
- d) Equations describing curves should be placed in the text, lengthy tabular material should be presented in a separate numbered table, and explanations and conditions should be added to the legends or placed in the text.
- e) Clearly label what is plotted on each axis and the units used. Whenever possible plot all parts of any one figure or related figures on scales with the same increments and range. For ease in interpolation divide scales into logical, consistent increments.
- f) Label main and auxiliary scales with a word description and its unit. For example, "Dissolved Oxygen Concentration (mg/l)" is more descriptive than "Oxygen". Add auxiliary scales at the left and bottom of the figure if there are four or fewer scales. Place additional scales at the right or top.
- g) Avoid un-necessary trailing zeros when labeling scales.
- h) Decide whether graphs will be in colour or black and white and choose markers and lines appropriately.
- i) Use the same data symbols and lines to represent the same conditions consistently throughout the graphs of the report.
- j) Keys are preferred when several curves are plotted on a single graph or when several conditions are associated with each curve. Keys generally follow the format for tabular material and should be consistent throughout a set of figures.

4.2.4 Drawings

When including drawings or maps to illustrate sampling locations, pollution sources or sensitive receivers keep them simple and comply with the following guidelines:

- a) Include only those features in the drawing that are essential for the readers' understanding. Avoid unnecessary detail.
- b) Decide whether drawings will be in colour or black and white and prepare the drawing accordingly.
- c) Avoid un-necessary shading and rendering on the drawing.

4.2.5 Photographs

The need for high quality color printing should be considered carefully because it greatly increases printing costs. Photographs that are clear when seen in colour, in many cases, will lose much of their value when printed in black and white.

Include some object or scale in the photograph to help the readers judge the size of the objects shown.

4.2.6 Tables

Tables are often included in reports to present data in an exact, highly concentrated form. Because tabulated data are so concentrated, the reader may have difficulty in absorbing all of the information. Tables in the body of the text are therefore the least preferred method of presenting results. They should be used only when absolutely necessary.

Tables should be as brief and simple as possible. Otherwise the reader may not spend time studying the concentrated columns of figures. If the information in the table can be put into words it is better to do so.

Figure 4.1 shows a typical layout of a table whose purpose is to show the changes in dissolved oxygen, BOD, ammonia and the GQA index at stations along the river Blackwater with the stations ordered from upstream to downstream.

Tables are numbered in the order in which they are mentioned using Arabic numerals. Similar data at different conditions are organized into parts (a), (b), (c), etc. of the same table with subtitles. Numbered tables must have title.

Numbers that are to be compared should be placed in columns.

Table 1. Statistics for dissolved oxygen, BOD and ammonia in the Blackwater river and the resulting GQA index

					←	Table caption
Station	Dissolved Oxygen (%sat) 10%ile	BOD (mg/l) 90%ile	NH ₃ ⁺ (mgN/l) 90%ile	GQA* index	←	Column titles
RB01	85	3.1	0.15	B		
RB02	76	4.2	0.18	C		
RB03	93	2.8	0.10	B		
RB03	87	2.9	0.11	B	←	Table Body
RB05	96	2.1	0.09	A		
Hendon weir	91	2.0	0.06	A		
*GQA is defined by worst performing determinand					←	Footnotes
Ref: Thames Tributaries Water Quality. 2010. Environment Agency. HMSO.					←	Source Notes

Figure 4.1 Components of a table and an example layout

Table caption: The main heading must describe the contents of the table and indicate the purpose of the table. The format of the title is generally bold sentence case.

Column titles: The vertical heading and subheadings of the columns are known as column titles. Generally only the first letter of the box head is in capital letters and the remaining words are in lower case.

It is normal to use the columns for variables whose values vary – such as values measured or calculated

Row titles The horizontal headings and sub heading of the row are the row captions. As in the case of the column titles the caption is in capital letters and the remaining words are in lower case.

Rows are used for variables whose values do not change such as sampling locations and years.

The Body: the main part of the table that contains the numerical information classified with reference to the row and column captions.

Foot Notes: appear immediately below the body of the table providing explanation of a caption or values within the body of the table.

Source Notes: are given at the end of the table indicating the source of information that has been provided by a third party. It includes the information about compiling agency, publication etc...

Lines: Lines dividing the table into part should be used carefully and consistently through the report. The lines, together with spacing should be used to guide the eye of the reader through the table so that they see what you want them to see.

4.2.7 Placement of tables and diagrams

Graphs, summary tables, figures and diagrams should be used in the results and discussion section. Complex tables of raw data should be placed in an Appendix.

Tables, figures and graphs should be:

- a) Numbered sequentially
- b) Labelled clearly and
- c) Positioned as close to the associated text as possible, usually following the first reference to them.

4.2.8 References to figures and tables

Tables of data are quite clearly referred to as tables. However, for drawings, maps, graphs, figures and photographs there is no rigid naming scheme. It may be defined in the internal or external 'house style', and when writing for external publications the editor will provide their 'house style' which should be followed. In the absence of a pre-defined system consistency within the report is essential. A suggested scheme is as follows:

- a) Graphs, histograms, bar charts and pi-charts – referenced as Figures.
- b) Maps and drawings – referenced as Drawings.
- c) Photographs – referenced as Photographs.

Make specific reference to each figure, graph and table. Do not assume that the reader will make the necessary connection between the text and the figure or table. A direction look at each figure and table is required.

Refer to each figure, drawing, photograph or table in the text by its number.

When referring to tables and figures, phrases such as the following can be used:

‘As shown in Table I below, pH values were between 7.8 and 8.4’

‘Mean concentrations of Nitrate-nitrogen are shown in Table 2.2’

‘During the year the measured BOD concentrations frequently exceeded 8mg/l (Figure 3.1).

4.2.9 Numbering and labeling

If there are a large number of tables and figures in a report the placing of additional ones into the early sections of the report will require complete re-numbering of all of the following tables and figures. It is more convenient to include the section number as part of the table of figure number. For example the fourth table in section six of the report will be numbered Table 6.4. Any changes in sections one to five will have no effect on the table number.

As a general rule, labels for tables are placed above the table and labels for figures below the figure.

The label for a table or figure from a third party should contain an acknowledgement within the label like any other outside information and its source should be provided in the list of references.

All tables and figures should be labeled, even if the report contains only one.

Keep labels brief but informative. Explanatory notes, if they are needed, can be placed as footnotes under the table or figure.

MS Word provides tools for automatically numbering labels for tables and figures which will automatically renumber if new tables or figures are inserted or are moved to different places in the report. In the same way that numbered chapter and section headings can be automatically collated into a table of contents lists of figures and tables can be inserted in the table of contents section of the report.

The tool is accessed through the **Insert/Captions** dropdown menu in MS Word and the creation of the list through the **Insert/Index and Tables** menu.

4.2.10 Figures and tables in appendices

Number figures and tables in appendices according to the appendix in which they appear. For example in Appendix A you would have Figure A1, Table A1.

A list of the figures and tables contained in each appendix should either be part of or follow the ToC for the Appendix, not following the ToC for the main report.

4.2.11 General Layout

Solid blocks of text are both very tiring to read and are likely to transfer information to the reader less effectively than more distributed text. Break up the text using the following techniques such as:

- a) Use of wide margins;
- b) Spacing out of paragraphs; and
- c) Style and indent of subheadings.

The use of the MS Word tool **STYLES** will ensure consistency in font and layout through out the document. This is particularly valuable when many authors are involved in writing the text and will save much time at the final editing stage.

Following the planning stage for the report prepare a template report containing all of the permitted styles with the agreed section headings included. This will ensure consistency of physical style between different authors and greatly speed up the final compilation and editing of the report.

5 Finalisation of the Report

5.1 Peer Review

Familiarity with a report tends to blind the reader to small mistakes. Even if the Quality Management System does not require the document to be checked before being distributed consider asking a colleague to read it through.

5.2 Checklist

A checklist is a useful tool for carrying out the final sweep of the report and ensures that all parts of the document including the appendices have been compiled correctly added either electronically or physically.

Before the report is submitted for approval it is important to ensure that the report:

- a) Fulfills its objectives,
- b) Follows a logical structure,
- c) Is complete with regard to tables, figures appendices etc
- d) Has no spelling or grammatical errors.

Typical elements of a checklist are given below.

Content

- ☐ Are the purpose and aims of the report clearly set out?
- ☐ Are readers needs taken into account?
- ☐ Are the main points included?
- ☐ Are the points supported by referenced evidence?

- ☐ Is all the information relevant to the purpose?
- ☐ Are the conclusions and recommendations clearly linked to the purpose of the report and based on findings that have been presented?

Structure

- ☐ Is there any (unnecessary) repetition?
- ☐ Is the order logical?
- ☐ Are the headings and numbering clear?
- ☐ Is the information presented clearly?
- ☐ Is there a good use of graphics?
- ☐ Are all the tables and figures referenced and visa versa
- ☐ Is the language clear and easy to understand?
- ☐ Is the style formal?
- ☐ Is the tone suited to the purpose?
- ☐ Are the any unnecessary words or phrases?
- ☐ Is the grammar and punctuation correct?
- ☐ Is the spelling correct?

Compilation

- ☐ Are all the sections present?
- ☐ Are all the appendices present?

Bibliography:

Guidelines for preparing statistical tables and reports. Tennessee Department of Health Policy, Planning and Assessment. Division of Health Statistics. January 2006.

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Rudd, D., Report Writing. A guide to organisation and style. Learning Support and Development. University of Bolton. UK. 2005.

<http://www.bolton.ac.uk/library/LibraryPublications/StudySkills/reportw.pdf>

Report Writing. A take-away guide. Multimedia Publishing Ltd, 1975

<http://lorien.ncl.ac.uk/tskills/reports/repwrite.pdf>

Almost Everything You Wanted to Know About Making Tables and Figures.

http://abacus.bates.edu/~ganderso/biology/resources/writing/HTW_Guide_Table-Figures_9-30-08.pdf

Appendix A

Example structures for factual, instructive and leading reports.

A1: Example Structure for a Factual Report

Annual Water Quality Report

1 Introduction

2 Background

2.1 Historic water quality

2.2 Trends in water quality

2.3 Sources of pollution

2.4 Impacts on beneficial uses

2.5 Measures in place to minimize impacts

3 Methods and Materials

3.1 Locations and characteristics of sampling locations

3.2 Timetable of sampling

3.3 Field procedures

3.3.1 Sampling

A. Bankside

3.3.2 Bridge

B. Boat

3.3.3 *In-situ* measurements

A. Dissolved oxygen

B. Temperature

C. pH

D. Aesthetic

3.3.4 Sample preservation

3.4 Laboratory procedures

3.4.1 Physical determinands

A. Conductivity

B. Turbidity

C. Suspended solids

D.

3.4.2 Organo-leptic

- A. Colour
- B. Odour

3.4.3 Inorganic determinands

- A. Nitrogen
 - a. Nitrate
 - b. Nitrite
 - c. Ammonia
 - d. Organic nitrogen
- B. Phosphorus
 - a. ortho-phosphate
 - b. total phosphate
- C. Silica
- D.

3.4.4 Heavy metals

- A. Cadmium
- B. Copper
- C. Lead
- D. Mercury
- E. Nickel
- F.

3.4.5 Organic determinands

- A. BOD
- B. Loss on ignition
- C.

3.4.6 Trace organic compounds

- A. Herbicides
 - a. Atrazine
 - b. Diuron
 - c. Symazine
 - d.
- B. Pesticides
 - a. Diazinon
 - b. Methoxychlor
 - c.

3.4.7 Microbiological

- A. Faecal streptococci
- B. E coli
- C.

3.5 Quality control procedures

3.6 Statistical analysis

3.6.1 Excel

3.6.2 AARDVARK

4 Results and discussion

4.1 River Kennet

4.1.1 Physical determinands

- E. Conductivity
- F. Turbidity
- G. Suspended solids
- H.
- I. Discussion of compliance, trend etc

4.1.2 Organo-leptic

- C. Colour
- D. Odour
- E. Discussion of compliance, trend etc

4.1.3 Inorganic determinands

- E. Nitrogen
 - a. Nitrate
 - b. Nitrite
 - c. Ammonia
 - d. Organic nitrogen
 - e. Discussion of compliance, trend etc

Carry on to include all of the groups described in Section 3

4.2 River

5 Conclusions

5.1 Introduction

5.2 Overall monitoring plan

5.3 Compliant determinands

5.4 Non-compliant determinands

References

Appendix A - Details of sampling stations
Appendix B – Equipment list
Appendix C – List of laboratory methods used
Appendix D – Tables of monthly results
Appendix E – Tables of QC results
Appendix F – List of water quality standards

A2: Example Structure for an Instruction Report

Measurement of marine water BOD

Cover page to include standard controlled document block.

1 Introduction

2 Materials

2.1 Equipment

2.1.1 Refrigerator

2.1.2 Incubation bottles

2.1.3 Other glassware

2.1.4 Sample holding racks

2.1.5 Balance

2.1.6 Pipettes

- A. Manual
- B. Piston Dispensing

2.1.7 Dissolved oxygen meter and probe

2.1.8 Air pump and diffuser stones.

2.1.9 Incubator

- A. Temperature fluctuation checks
- B. Internal temperature distribution checks

2.2 Reagents

2.2.1 ATU

2.2.2 Standards

2.2.3 Result recording sheets

3 Method

3.1 Preparation of standard addition solution

3.2 Calibration of dissolved oxygen probe

3.3 Warming/cooling of sample to 20 deg C

3.4 Measurement of initial dissolved oxygen concentration.

3.5 Raising to saturation concentration

3.6 Preparation of blank samples

3.7 Preparation of replicate samples

3.8 Preparation of standard addition samples

3.9 Measurement of T_0 oxygen concentrations

3.10 Daily incubator temperature check and inspection

3.11 Measurement of T_5 oxygen concentrations

3.12 Washing and storage of incubation bottles

3.13 Calculations

3.14 Evaluation of QC data.

3.15 Actions in the event of QC failure

3.16 Reporting

4 Health and safety requirements

4.1 Health and safety equipment

4.2 Disposal of samples and reagents

5 References.

A3: Example Structure for a Leading Report

Proposal for more effective management of pollution to the River Towy by leather tanning industries

1 Introduction

2 Problems caused by untreated tanning wastewater

2.1 Introduction

2.2 BOD

2.3 COD

2.4 Chromium

3 Sections of the river affected by the waste water

3.1 Locations of industries

3.2 Locations and types of sensitive receivers

4 Current level of pollution management

4.1 Inventory of industries

4.2 Inspection and effluent monitoring program

4.3 River water quality monitoring

5 Pollution load analysis

5.1 Present chromium loads to the river from industries

5.2 Assimilation capacity of the river

5.3 Maximum acceptable chromium load to the river

6 Options for reducing chromium load

6.1 Introduction

6.2 Regulatory Intervention

6.3 Technological Intervention

6.3.1 Individual industries

6.3.2 Grouped industries

6.4 Implementation scenarios.

6.4.1 Capital cost implications

6.4.2 Recurrent cost implications

7 The do nothing scenario

7.1 Human health consequences and costs

7.2 Ecological consequences and costs

8 Recommendations

Appendix A – Details of industries involved

Appendix B – Water and sediment analysis

Appendix C – Results of fish and invertebrate surveys

Appendix D - Acute and chronic eco-toxicity data for chromium

Appendix E- Pollutant mass balance model

Appendix F – Analysis of the environmental value of the river

Appendix G – Cost-benefit analysis of interventions

APPENDIX-5

***DRAFT MANUAL FOR WATER QUALITY DATA
MANAGEMENT SYSTEM
IN THUA THIEN-HUE, VIETNAM
FEBRUARY, 2012***

SCOWEM

*Project for Strengthening
Capacity of Water Environmental Management in
Vietnam*

DRAFT

MANUAL

FOR

WATER QUALITY DATA

MANAGEMENT SYSTEM

IN

THUA THIEN – HUE, VIETNAM

FEBRUARY, 2012

THUA THIEN – HUE DONRE

&

JICA EXPERT TEAM

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2 Database Structure	1
3 Data flow	3
4 Database Format	4
4.1 Regular Monitoring Program	4
4.2 Database Format.....	4
5 Database Management and Backup	9
5.1 Database Management	9
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1 Introduction

This manual shows the data management method in Water Quality Data Management System (hereinafter referred to as “WQDMS”), as a part of procedure to improve the capacity of water environmental management in Thua Thien – Hue Environmental Protection Agency (hereinafter referred to as EPA) under Department of Natural Resources and Environment (hereinafter referred to as DONRE) in Thua Thien – Hue province, Vietnam.

The purposes of WQDMS are one of the activities for Quality Assurance and Quality Control (QA/QC). The objective of QA/QC is to have clear and concise records of the procedures which may have a bearing on the quality of data. Especially from the view points of data management, it is very important for managing agency to secure traceability of analyzed data which then will be converted to information provided to the public.

In addition, this data management manual is prepared to enable WQDMS staffs to manage the database properly in their daily works and also shows staffs their roles of the data management. This manual provides necessity information for the database manager of monitoring section of EPA. Though this manual is mainly written for the staff that has a responsibility for the update and maintenance of water quality database, the other staffs also necessity follow this document.

2 Database Structure

There are four (4) main categories of water quality in WQDMS; 1) Surface water, 2) Groundwater, 3) Wastewater and 4) Coastal water. In each category, there are several types of water quality data. From 2010, all of water samples are taken quarterly and reported every six months.

Table 2-1: Categories of monitoring data

Category	Water quality data – Number of sampling points
1) Surface water	1) River – 28 2) Lakes - 02
2) Groundwater	1) Groundwater - 20
3) Wastewater	1) Huong river outfalls - 04 2) Textile, dyeing, paint, pesticide industries - 02 3) Other type of industrial wastewater - 05 4) Food processing - 06 5) Shrimp farming and slaughtering – 03 6) Restaurants, Landfill sites, WWTPs - 05
4) Coastal water	1) Lagoon - ?

In accordance with monitoring type, the water quality database has been established. Figure 2-1 shows the database structure of water quality database and its contents.

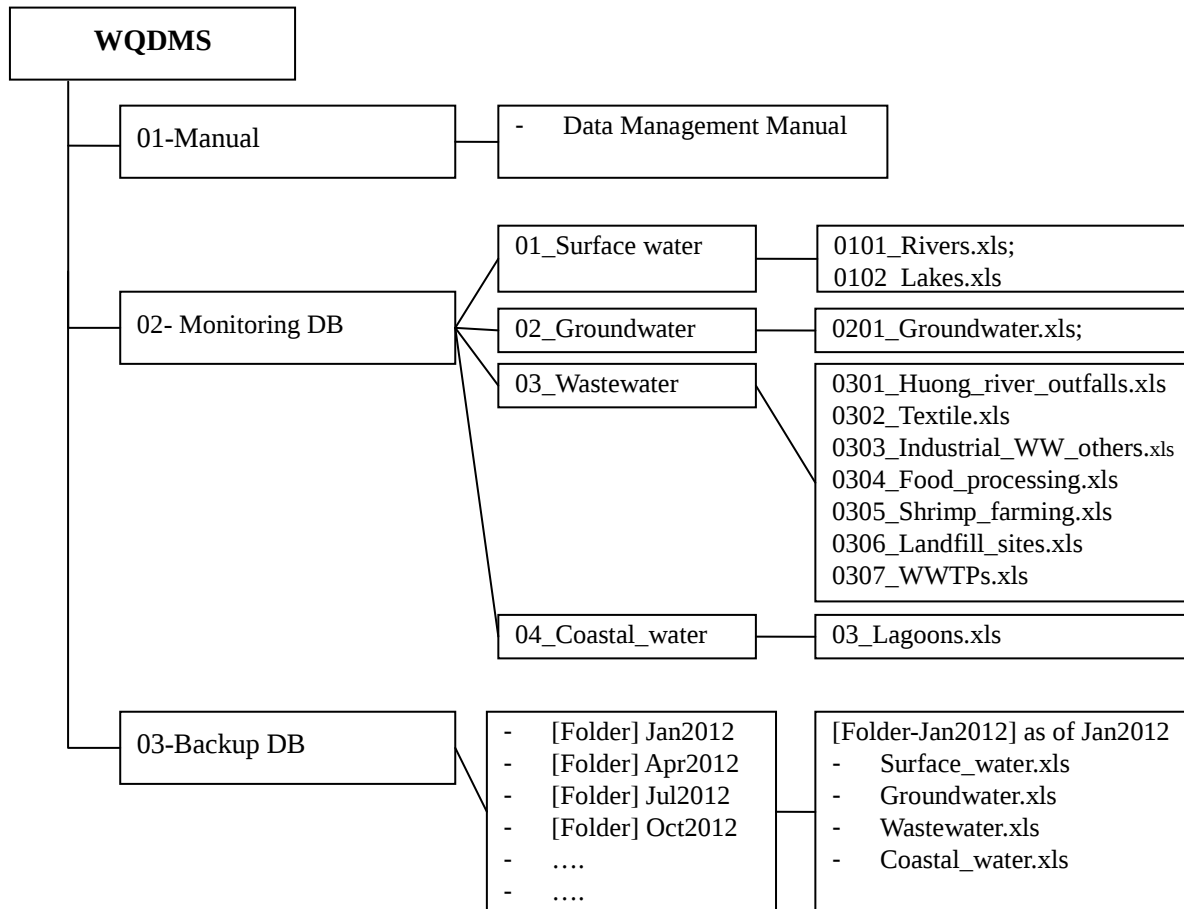


Figure 2-1: Structure of WQDMS

Table 2-2: Directories of monitoring data

Directory	Content
01-Manual	01 Manual - Data Management Manual
02-Monitoring DB	The latest 4 folders. Based on categories of monitoring data: - 01_Surface_water - 02_Groundwater - 03_Wastewater - 04_Coastal_water
03-Backup Database	create a backup folder four times a year (Detailed description is in the chapter “Data backup”)

3 Data flow

Figure 3-1 shows the data flow in WQDMS, and also shows the staffs in charge in each step of the data flow.

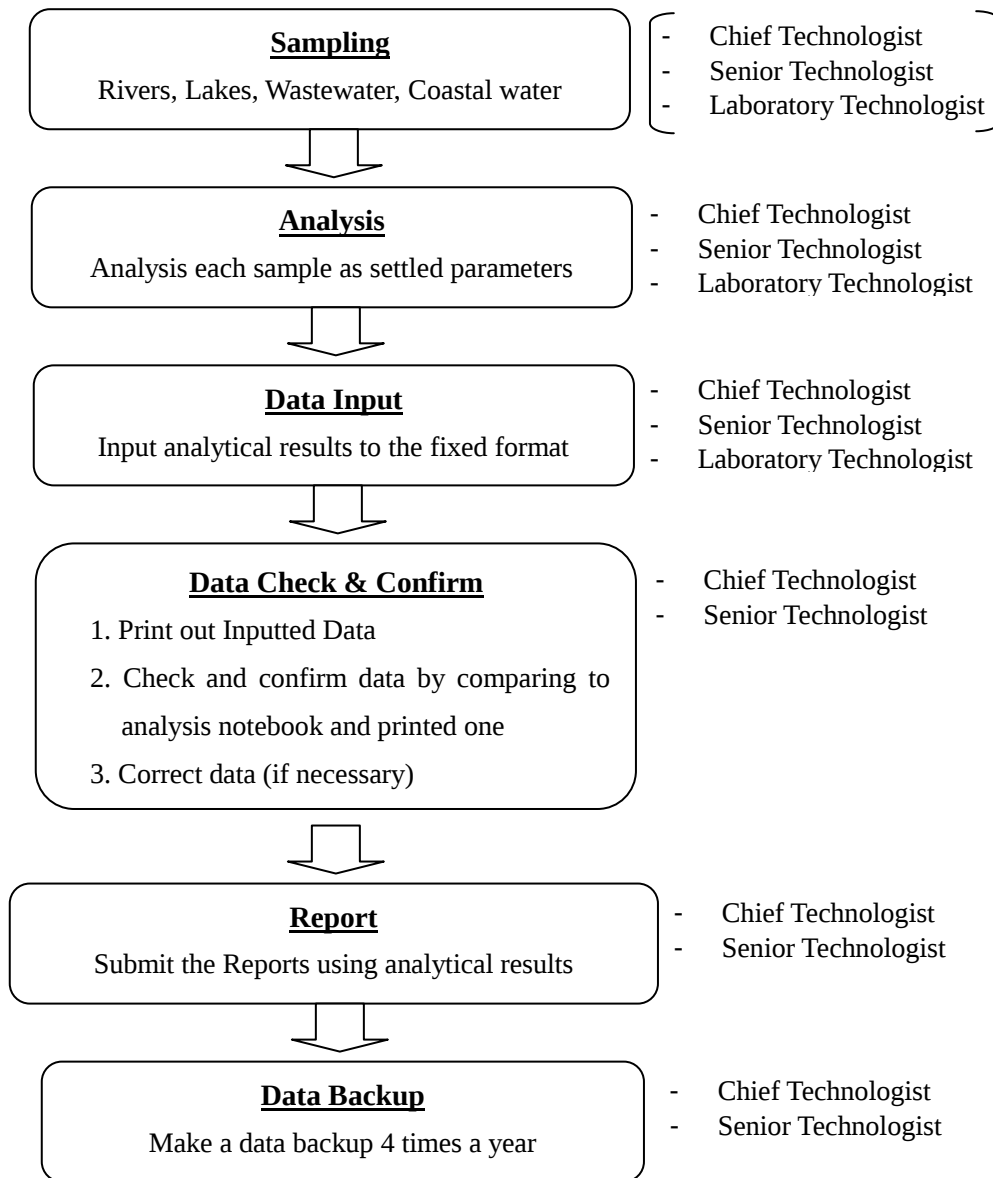


Figure 3-1: Data Flow and Responsible Staff in WQDMS

4 Database Format

4.1 Regular Monitoring Program

The regular monitoring plan is re-assessed and revised annually in case of necessity. Based on this revised plan, the format of the water quality database is also revised. The revised monitoring program including new settled sampling point, analytical parameter of each sampling point and its frequency of sampling.

When the regular monitoring plan will be revised in the future, data format will also be revised in accordance with the revised program.

4.2 Database Format

The database of WQDMS is composed of the four (4) folders, each folder contains one or several excel files depending on the type of monitoring data as shown in Figure 2-1.

Each excel file has a same format. The detailed format of database is shown in Figure 4-1

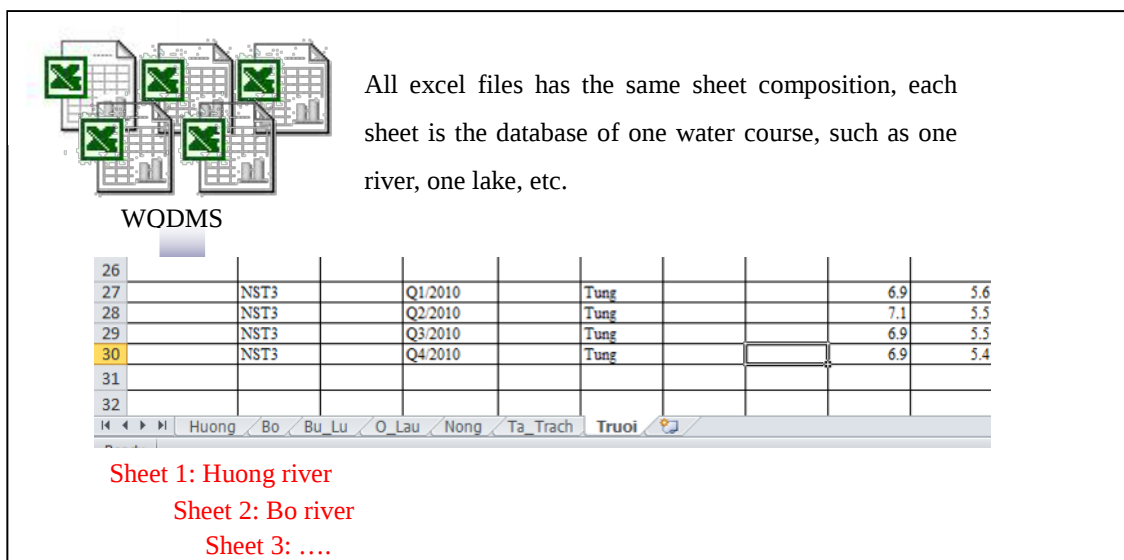


Figure 4-1: Database Sheet Composition

[Code of monitoring point]

In Thua Thien – Hue, monitoring points are coded in the following format: N_[Name-of-water-course]_[Number] or N_[Name-of-wastewater]_[Number] as shown from Table 4-1 to Table 4-4.

Table 4-1: Sampling Point Code of Surface water

Water course	Code	File name	Sheet name
Huong river	NSH1	0101_Rivers.xlsx	Huong
	NSH2	0101_Rivers.xlsx	Huong
	NSH3	0101_Rivers.xlsx	Huong
	NSH4	0101_Rivers.xlsx	Huong
	NSH5	0101_Rivers.xlsx	Huong
	NSH6	0101_Rivers.xlsx	Huong
	NSH7	0101_Rivers.xlsx	Huong
	NSH8	0101_Rivers.xlsx	Huong
Bo river	NSB1	0101_Rivers.xlsx	Bo
	NSB2	0101_Rivers.xlsx	Bo
	NSB3	0101_Rivers.xlsx	Bo
	NSB4	0101_Rivers.xlsx	Bo
Bu Lu river	NSBL1	0101_Rivers.xlsx	Bu_Lu
	NSBL2	0101_Rivers.xlsx	Bu_Lu
	NSBL3	0101_Rivers.xlsx	Bu_Lu
O Lau river	NSOL1	0101_Rivers.xlsx	O_Lau
	NSOL2	0101_Rivers.xlsx	O_Lau
	NSOL3	0101_Rivers.xlsx	O_Lau
	NSOL4	0101_Rivers.xlsx	O_Lau
Nong river	NSN1	0101_Rivers.xlsx	Nong
	NSN2	0101_Rivers.xlsx	Nong
	NSN3	0101_Rivers.xlsx	Nong
Ta Trach river	NSTT1	0101_Rivers.xlsx	Ta_Trach
	NSTT2	0101_Rivers.xlsx	Ta_Trach
	NSTT3	0101_Rivers.xlsx	Ta_Trach
Truoi river	NST1	0101_Rivers.xlsx	Truoi
	NST2	0101_Rivers.xlsx	Truoi
	NST3	0101_Rivers.xlsx	Truoi
Huong		0102_Channels.xls	Huong
Thuong Tu		0102_Channels.xls	Thuong_Tu
Dong Ba		0102_Channels.xls	Dong_Ba
Hau		0102_Channels.xls	Hau
An Hoa		0102_Channels.xls	An_Hoa
Chanh Tay		0102_Channels.xls	Chanh_Tay
Tinh Tam lake	NHTT	0103_Lakes.xlsx	Tinh_Tam
Chau Son lake	NHCS	0103_Lakes.xlsx	Chau_Son

Table 4-2: Sampling Point Code of Groundwater

Area	Code	File name	Sheet name
Hue city	NGH1	0201_Groundwater.xlsx	TP Hue
	NGH2	0201_Groundwater.xlsx	TP Hue
	NGH3	0201_Groundwater.xlsx	TP Hue
	NGH4	0201_Groundwater.xlsx	TP Hue
	NGH5	0201_Groundwater.xlsx	TP Hue

Area	Code	File name	Sheet name
Phong Dien district	NGPD1	0201_Groundwater.xlsx	Phong Dien
	NGPD2	0201_Groundwater.xlsx	Phong Dien
Quang Dien district	NGQD1	0201_Groundwater.xlsx	Quang Dien
	NGQD2	0201_Groundwater.xlsx	Huong Thuy
Huong Tra district	NGHT1	0201_Groundwater.xlsx	Huong Tra
	NGHT2	0201_Groundwater.xlsx	Huong Tra
Huong Thuy district	NGHTh1	0201_Groundwater.xlsx	Huong Thuy
	NGHTh2	0201_Groundwater.xlsx	Huong Thuy
Phu Loc district	NGPL1	0201_Groundwater.xlsx	Phu Loc
	NGPL2	0201_Groundwater.xlsx	Phu Loc
Phu Vang district	NGPV1	0201_Groundwater.xlsx	Phu Vang
	NGPV2	0201_Groundwater.xlsx	Phu Vang
A Luoi district	NGAL1	0201_Groundwater.xlsx	A Luoi
	NGAL2	0201_Groundwater.xlsx	A Luoi
Nam Dong district	NGAD1	0201_Groundwater.xlsx	Nam Dong
	NGAD2	0201_Groundwater.xlsx	Nam Dong

Table 4-3: Sampling Point Code of Wastewater

Type of wastewater	Code	File	
Huong river outfalls	NDT1	0301_Huong_river_outfalls.xlsx	DoThi
	NDT2	0301_Huong_river_outfalls.xlsx	DoThi
	NDT3	0301_Huong_river_outfalls.xlsx	DoThi
	NDT4	0301_Huong_river_outfalls.xlsx	DoThi
Textile	NTD1	0302_Textile.xlsx	CP_Det_Hue
	NDT1	0302_Textile.xlsx	CP_Det_Hue
Industrial wastewater (others)	NTIn1	0303_Industrial_WW_others.xlsx	CP_In_TTHue
	NTIn2	0303_Industrial_WW_others.xlsx	CP_In_TTHue
	NTCS1	0303_Industrial_WW_others.xlsx	CP_Caosu_TTHue
	NTCS2	0303_Industrial_WW_others.xlsx	CP_Caosu_TTHue
	NTDL1	0303_Industrial_WW_others.xlsx	KS_DongLoi
	NTDL2	0303_Industrial_WW_others.xlsx	KS_DongLoi
Food processing	NTGM1	0304_Food_processing.xlsx	NNSS_TTHue
	NTGM2	0304_Food_processing.xlsx	NNSS_TTHue
	NTBS1	0304_Food_processing.xlsx	Botsan_Fococev
	NTBS2	0304_Food_processing.xlsx	Botsan_Fococev
	NTTS1	0304_Food_processing.xlsx	PT_Thuysanb_TTHue
	NTTS2	0304_Food_processing.xlsx	PT_Thuysanb_TTHue
Shrimp farming		0305_Shrimp_farming.xlsx	
Landfill sites		0306_Landfill_sites.xlsx	
Wastewater treatment plant		0307_WWTPs.xlsx	

Table 4-4: Sampling Point Code of Coastal water

Area	Code	File name	Sheet name
Tam Giang lagoon	NPTG1	0401_Lagoons.xlsx	Tam_Giang
	NPTG2	0401_Lagoons.xlsx	Tam_Giang
	NPTG3	0401_Lagoons.xlsx	Tam_Giang
	NPTG4	0401_Lagoons.xlsx	Tam_Giang
	NPTG5	0401_Lagoons.xlsx	Tam_Giang
	NPTG6	0401_Lagoons.xlsx	Tam_Giang
Cau Hai lagoon	NDCH1	0401_Lagoons.xlsx	Cau_Hai
	NDCH2	0401_Lagoons.xlsx	Cau_Hai
	NDCH3	0401_Lagoons.xlsx	Cau_Hai
	NDCH4	0401_Lagoons.xlsx	Cau_Hai
	NDCH5	0401_Lagoons.xlsx	Cau_Hai
	NDCH6	0401_Lagoons.xlsx	Cau_Hai
	NDCH7	0401_Lagoons.xlsx	Cau_Hai
	NDCH8	0401_Lagoons.xlsx	Cau_Hai
Thuy Tu lagoon	NDTT	0401_Lagoons.xlsx	Thuy_Thu
Lang Co lagoon	N DLC1	0401_Lagoons.xlsx	Lang_Co
	N DLC2	0401_Lagoons.xlsx	Lang_Co
	N DLC3	0401_Lagoons.xlsx	Lang_Co
	N DLC4	0401_Lagoons.xlsx	Lang_Co

[Data structure]

Each sheet contains the water quality data and relevant data such as station name, station code, sampling date, parameters, standards and analyzed data. The sheet is divided into 3 main sections as shown in Figure 4-2

The screenshot shows a spreadsheet with the following structure:

- General information (rows 1-10):** Contains station details like Name (Mã địa điểm lấy mẫu), Station Code (Mã trạm), Sampling Date (Ngày lấy mẫu), Weather (Điều kiện thời tiết), and Name of data inputter (Nhập dữ liệu bởi) and data checker (Kiểm tra bởi).
- Parameters and standards (rows 11-18):** Lists water quality parameters (pH, Dissolved Oxygen, Turbidity, etc.) and their corresponding standards (QCVN 08-2008/HSTNM).
- Data records (rows 19-29):** Contains the actual measured data for each parameter across multiple sampling events.

Figure 4-2: Water quality datasheet

[1. General information]

General information section includes Name of water course; Code of station; Time and date of sampling; Weather condition; Name of data inputter and Name of data checker.

9																	
10		Trạm/Địa điểm lấy mẫu	Mã	Thời đoạn lấy mẫu	Ngày lấy mẫu	Điều kiện thời tiết	Nhập dữ liệu bởi	Kiểm tra bởi									
11																	
12		Sông Hương	NSH1		Q1+Q2/2009		Thái										
13			NSH1		Q3+Q4/2009		Thái										
14			NSH1		Q1/2010		Thái										
15			NSH1		Q2/2010		Tung										
16			NSH1		Q3/2010		Tung										
17			NSH1		Q4/2010		Tung										
18																	
19			NSH2		Q1+Q2/2009		Thái										
20			NSH2		Q3+Q4/2009		Thái										
21			NSH2		Q1/2010		Thái										

Figure 4-3: General information of water quality datasheet

[2. Parameters and standards]

The Parameters and standards section includes list of parameters being measured for that water course and limitation of respective parameters. Depending on the type of water and their respective QCVN, standards can be divided into only one or a few classes.

	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	AE	AZ
IT	1	2	3	4	5	6	7	8	9	10	11	12	13	23		
Tên chỉ tiêu	pH	Oxy hòa tan (DO)	Tổng chất rắn lơ lửng (TSS)	Độ đục	Độ dẫn điện	COD	BOD ₅ (20°C)	Amoni (NH ₄ ⁺) (tính theo N)	Clorua (Cl ⁻)	Fluorua (F ⁻)	Nitrit (NO ₂ ⁻) (tính theo N)	Nitrat (NO ₃ ⁻) (tính theo N)	Phosphat (PO ₄ ³⁻) (tính theo P)	Sắt (Fe)		Coliform
Đơn vị	-	mg/l	mg/l	FTU	mS/m	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	MPN/100ml
QCVN 08:2008/BTNMT (surface water)	6-8,5	≥ 6	20	-	-	10	4	0.1	250	1	0.01	2	0.1	0.5		2500
	6-8,5	≥ 5	30	-	-	15	6	0.2	400	1.5	0.02	5	0.2	1		5000
	5,5-9	≥ 4	50	-	-	30	15	0.5	600	1.5	0.04	10	0.3	1.5		7500
	5,5-9	≥ 2	100	-	-	50	25	1	-	2	0.05	15	0.5	2		10000

Figure 4-4: Parameters and standards

[3. Data records]

The data records section includes water quality data from the labs. Data will be digitalized from analytical result sheets from the labs. Each row contains the analytical results of parameters of one (01) sampling occasion.

9	Trạm/Địa điểm lấy mẫu	Mã	Thời đoạn lấy mẫu	Ngày lấy mẫu	Điều kiện thời tiết	Nhập dữ liệu bởi	Kiểm tra bởi	TT	1	2	3	4	5	6	7	8	9
10								Tên chỉ tiêu	pH	Oxy hòa tan (DO)	Tổng chất rắn lơ lửng (TSS)	Turbidity	Electrical conductivity	COD	BOD ₅ (20°C)	Amoni (NH ₄ ⁺) (tính theo N)	Clorua (Cl ⁻)
11								Đơn vị	-	mg/l	mg/l	FTU	mS/m	mg/l	mg/l	mg/l	mg/l
12	Sông Hương	NSH1		01+02/2009		Thái			7.0	6.1	25	20	6.5	8	5.2		7.5
13		NSH1		03+04/2009		Thái			7.1	6.1	25	2	6.8	6.8	5.4	0.15	7.1
14		NSH1		01/2010		Thái			7.1	6.1	18	1	6.3	6.5	4.6		7.2
15									7.2	6.8	15	1	6.4	6.2	4.3		7.1
16									6.9	6.6	20	1	6.4	6.2	4.2		7.1
17									6.8	6.5	21	3	6.6	6.5	5.6		4.3
18																	
19									7.1	6.3	28	25	6.5	9.6	6.4		12.1
20									7.2	6.6	28	2	6.6	8.7	6.2		7.1
21									7.1	6.5	16	1	6.4	6.8	4.9		7.2
22									7.1	6.7	15	1	6.6	6.5	4.6		7.1
23									6.9	6.5	20	1	6.6	6.8	4.7		7.1
24									6.7	6.4	25	4	6.7	10.6	6.2		4.3
25																	

Data from sampling at NSH1 in
Huong river at first sampling
occasion of 2009

Figure 4-5: Water quality data records

One datasheet may include data from several sampling locations, thus when one wants to input a new record at one sampling location, use the insert function in Excel to insert a new row right under the latest sampling occasion.

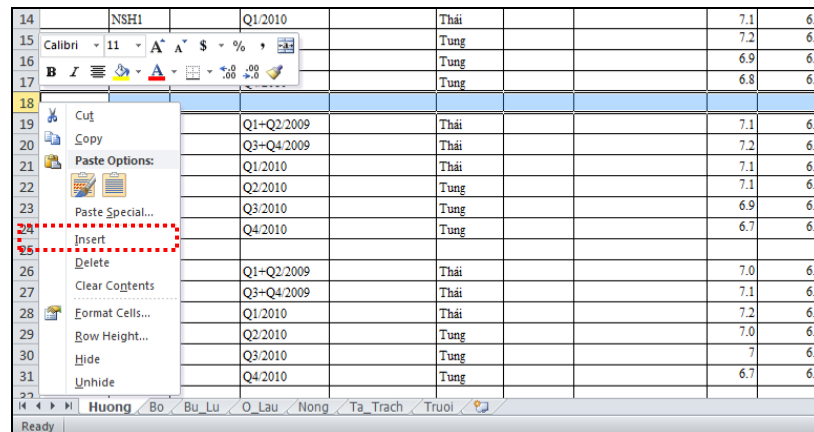
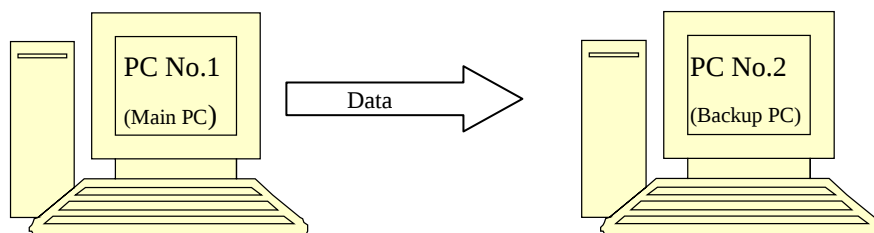


Figure 4-6: Insert a new record in datasheet

5 Database Management and Backup

The water quality database shall be managed with two (02) computers in the WQDMS. The desktop computer No.1 in Staff room is the main machine of WQDMS which will be used for the data management and data backup. Only desktop computer No.1 can be used for inputting checked data to avoid confusion. The desktop computer No.2 (Main User: Mr.X) is used for backup. The rule of data management in WQDMS is defined in Chapter 5.1.



5.1 Database Management

If the adjustment of the database cannot be taken in two computers, there are some possibilities that undesirable troubles occur such as repetition of data input, etc. It is also not undesirable system state from the viewpoint of database management. Therefore, the rules of data management in WQDMS are provided for proper data management.

- 1) A computer in Staff room No.1 is the main computer (hereinafter referred to as main PC) in WQDMS
- 2) Only staff in charge of WQDMS operation can access to the main PC (Set password)
- 3) The main PC can be used for daily tasks such as inputting checked data, reporting, and usage of pollution source inventory and inspection, record of environmental checking, GIS Database and GIS Analysis Module, etc.
- 4) Only checked data can be inputted into the main PC

- 5) The latest data is copied to Computer No.2 (Backup PC) as immediately as possible
- 6) The latest data is copied to the backup PC at latest at the same frequency as the data backup
- 7) In case that the main PC is not available (e.g. out of order), data can be inputted into the backup PC, and then the latest data is copied to the main PC immediately

5.2 Database Backup

There are two methods of backing up the database; 1) Save the latest database in the backup folder in a computer and an external hard disk and 2) a Make backup CD of the latest data, and then keep the backup CD in the shelf. The water quality database shall be backed up by these two methods. It is necessary to backup database regularly with both two methods and both two computers in monitoring section of EPA.

The backup of database executes four times a year, and the execution month refers to the following Table 5-1.

Table 5-1: Frequency of Database Backup

Jan	Feb	Mar	Apr	May	Jun	July	Aug	Sep	Oct	Nov	Dec
★			★			★			★		

★= Make Backup folder and CD

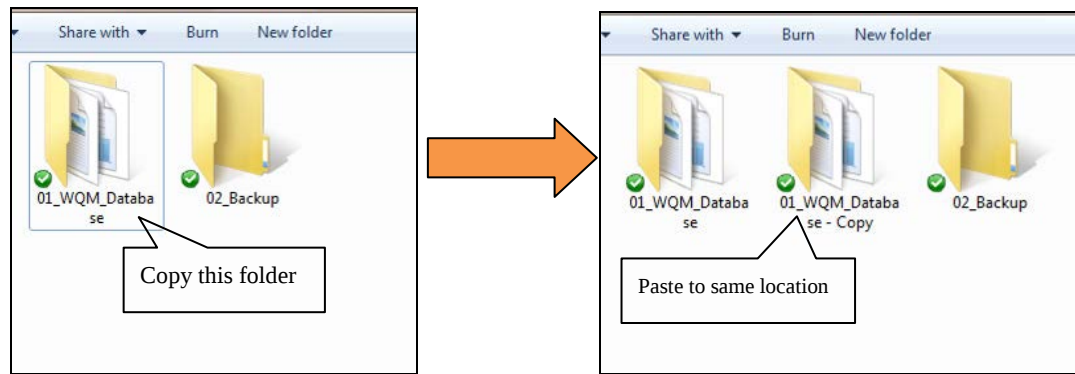
Each backup method is explained as follows.

5.2.1 Save the data into the backup folder

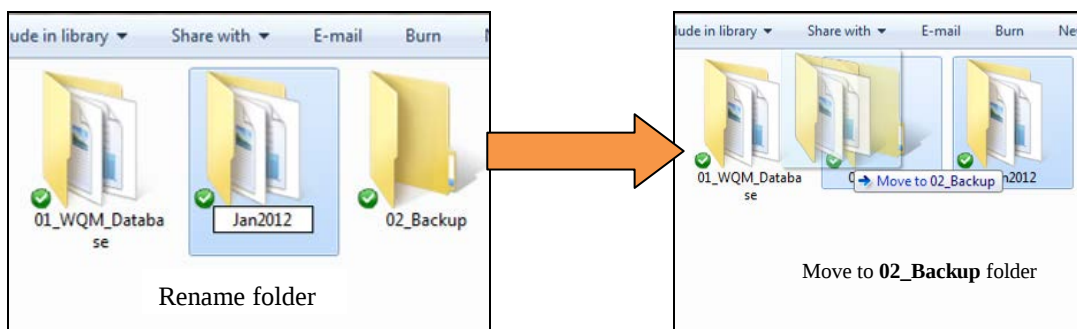
Every end of January, April, July and October, the data shall be backed up.

In this case, the name of the water quality monitoring folder is “**01_WQM_database**”, name of the backup folder is “**02_Backup**”.

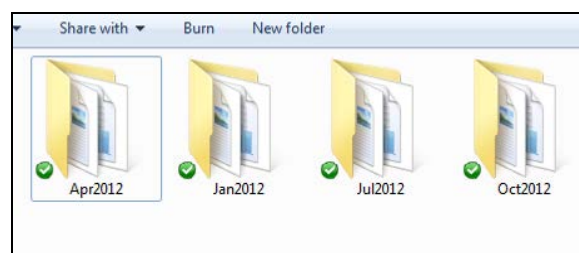
- First, copy the folder “**01_WQM_Database**” and paste to the same location, the copied folder namely “**01_WQM_Database –copy**” will be automatically created



- Rename the folder “**01_WQM_Database-copy**” to “**MonthYear**” when you create this backup, i.e. “**Jan2012**”
- Move (drag and drop) the renamed folder to “**02_Backup**” folder

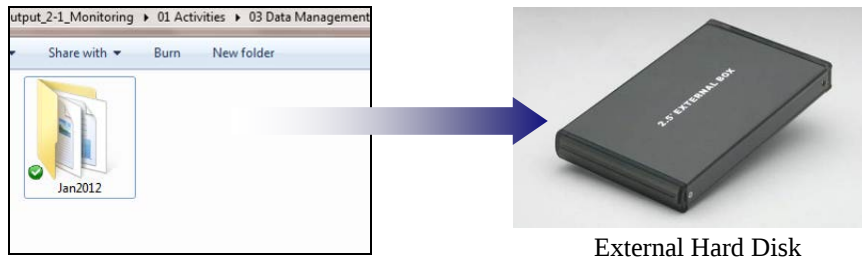


Following the same procedure, every four month, a new folder will be added to “**02_Backup**” folder



5.2.2 Transfer the backup folder to an External Hard Disk

There is an external-hard disk for data management in WQDMS. The backup folder is saved properly on an external hard disk at the same time as saving in the backup folder “02-Backup”.

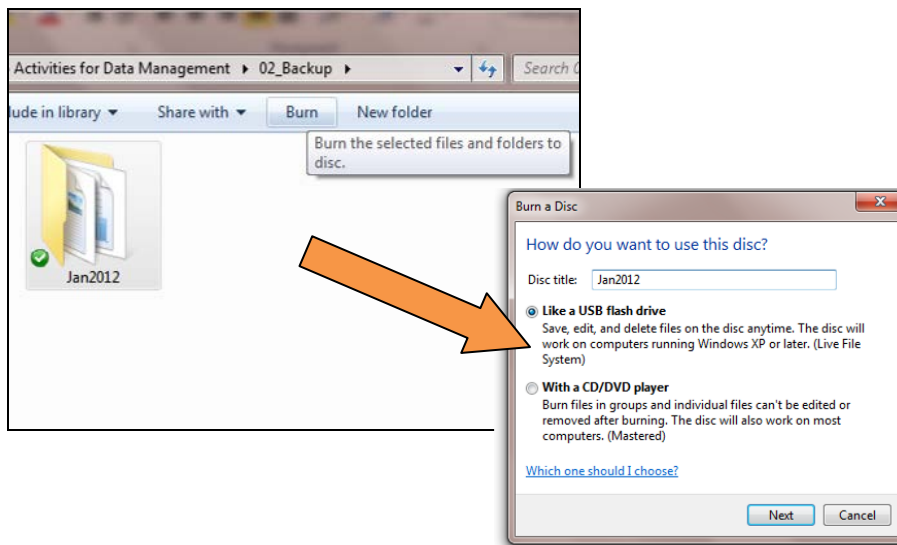


5.2.3 Create backup CDs

For the purpose of the further data management, regularly make backup CDs. CDs shall be made at the same timing as making the backup folder, it means 4 times a year. CDs can be created using a PC equipped with a CD writer.

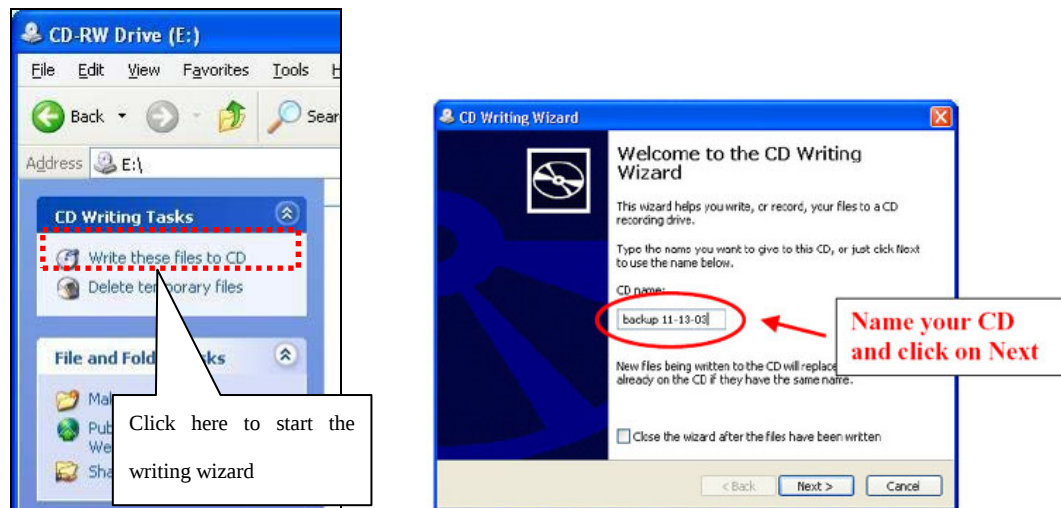
a) In Microsoft Windows Vista or Windows 7

- Insert a blank CD to the CD writer
- Select the folder and click “Burn”
- A new window will appear, name the CD the same as the folder.
- Select either “Like a USB flash drive” or “With a CD/DVD player”, depending on your needs.



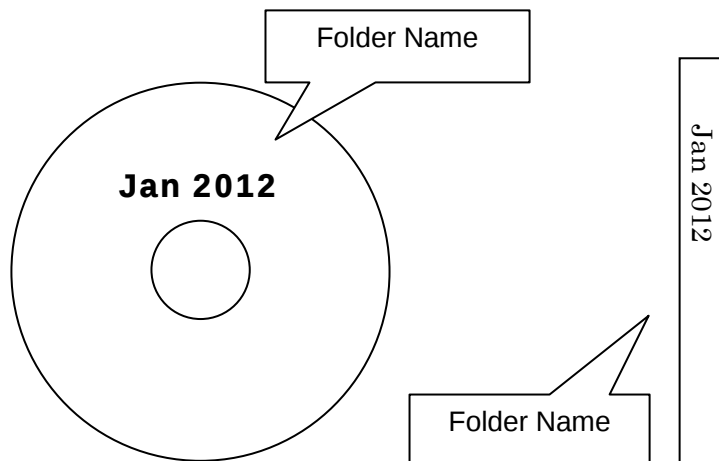
b) In Microsoft Windows XP

- Insert a blank CD to the CD writer
- Copy the folder to the CD drive, i.e. drive E:| as shown below
- From the common task menu, click “Write these files to CD”
- A new window will appear, Name your CD, i.e., Jan2012 and click Next



5.3 Rule of making label of CDs

The label of the newly created CD shall be applied. The rule of making the label is as follows.



5.4 Keep the backup CDs

The backup CDs shall be managed under the responsibility of Head of monitoring section in WQDMS. When the computer trouble occurs, the database shall be restored by using the data of this backup CDs.