Sampling analysis & Quality control

Based on presentations from Berend Lolkema, Bangladesh, 2019 & Fred Kruis, IHE)
(source picture: L. Strande 2014)

What is a sample?

How to choose your sampling strategy?

- What are the objectives of your study?
- What resources do you have?

  Money/Human/Time
- Other limitations

  Convenience/Accessibility

Types of sampling

1. Probability:

Each population member has an equal opportunity*of being in the sample

2. Non-Probability (Judgemental)*:*

Each population member does not have an equal opportunity* of being in the sample

* known probability

Advantages and disadvantages

	Probability	Non-Probability
Advantages	• Representative • Can generalise • Statistical inferences can be made	• Can be less expensive • Can be very efficient with knowledge • Easy to implement
Disadvantages	• Can be expensive • Can be difficult to design	• Depends on expert knowledge • Cannot generalise outside context • Depends on personal judgement to interpret data

Types of probability sampling

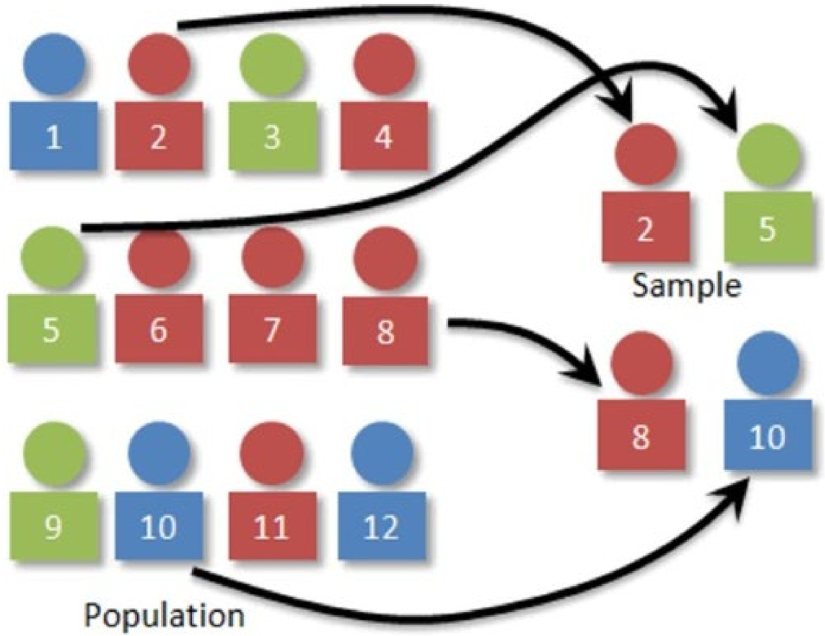
1. Random sampling
2. Stratified random sampling
3. Systematic sampling
4. Cluster sampling

Normally used when you want to make generalisations

1. Random Sampling

The members of the sample are selected randomly and purely by chance

- Random number tables
- Lottery system
- Random number generator



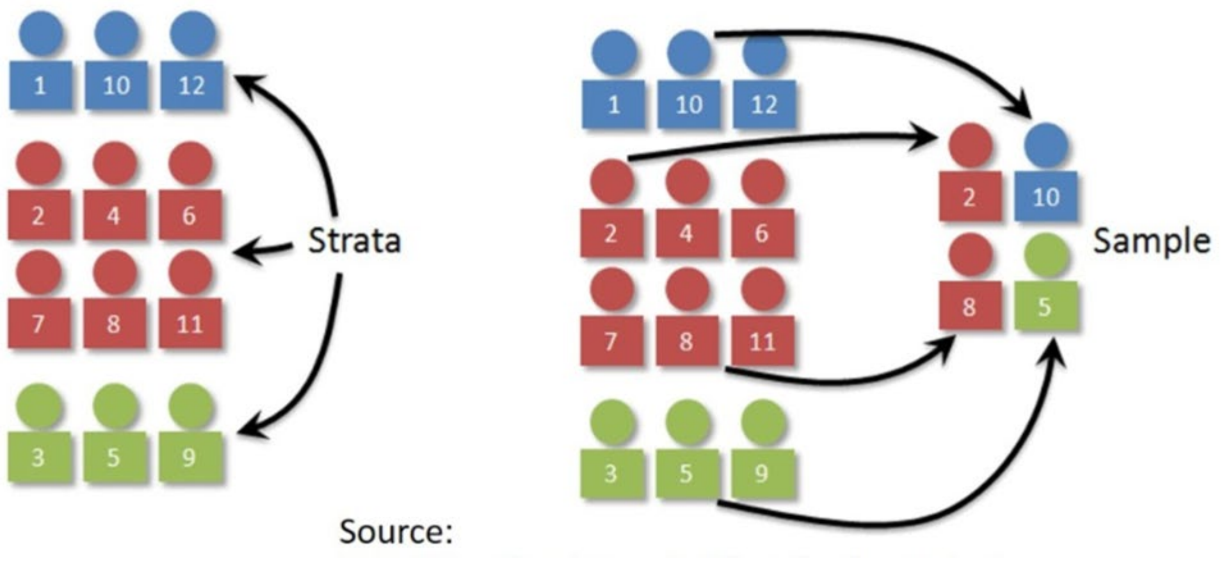
Aim of study:
To the know a the FS quality at Camp 13.

Randomly sample all septic tanks and latrines in Camp 13. Put toilet number in a hat and draw out the appropriate number – calculated using online tool

Used: When a population is highly homogenous

2. Stratified Random Sampling

1. The population is divided into sub-groups (*strata*)
2. The random sample is drawn from each sub-group randomly



Aim of study:

To understand the difference at different layers in pit latrines.

I take a core sample from a pit latrine, samples are taken at each horizon randomly.

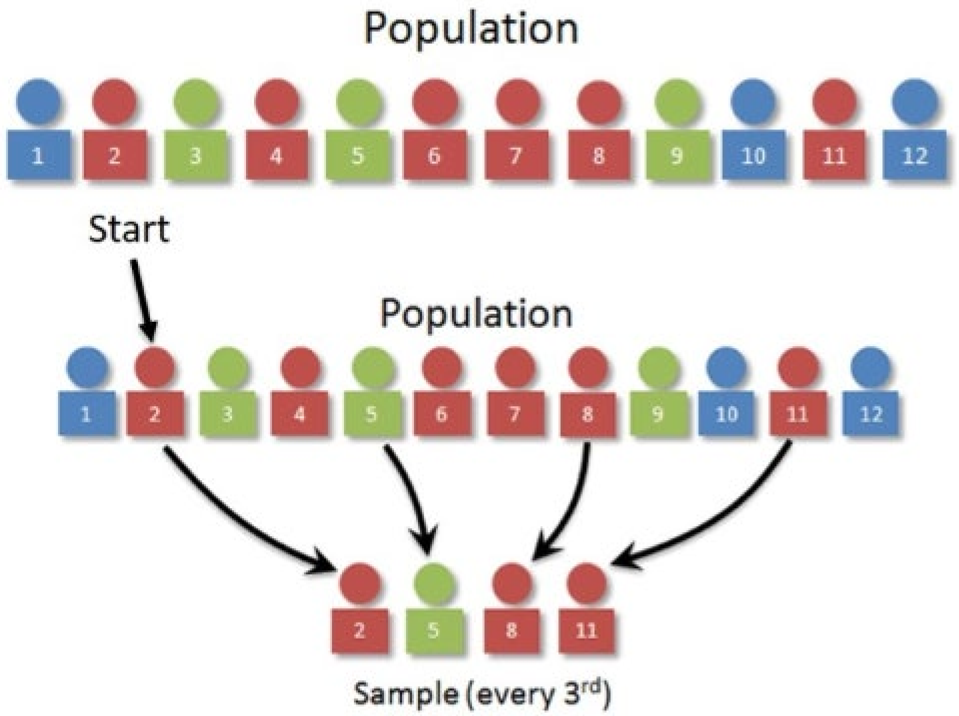
You might use another sampling strategy to select the pit latrines

Used: Population heterogeneous (divided into homogenous strata)

3. Systematic Sampling

A member after a fixed interval is selected

- Every 5th house
- Every hour
- Every 3 grid square
- Every 20 meters

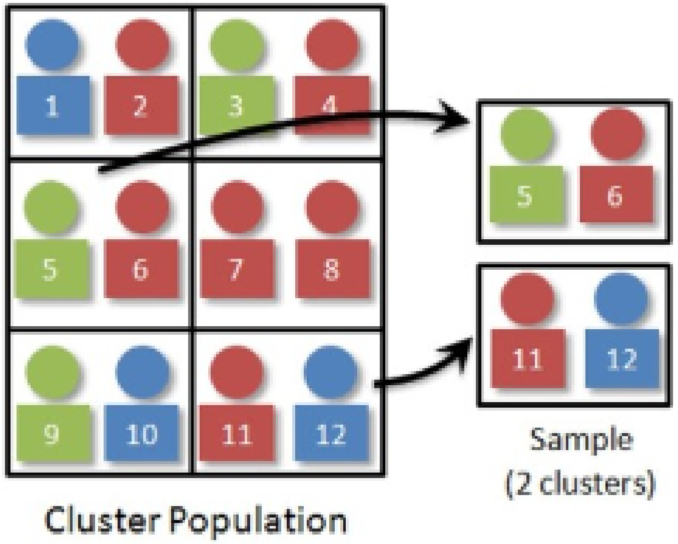


Aim of study:
You want to know the effluent quality of your treatment plant over 1 year (to look for seasonality).

You take a sample once a week and analyse it.

4. Cluster Sampling

1. Various segments of the population are treated as clusters.
   - Clusters are naturally formed units i.e. households
2. Clusters are randomly selected
3. Members from the chosen clusters can be selected randomly selected



What is the difference between stratified and cluster sampling?

Types of ***non-probability*** sampling

1. Convenience sampling

2. Purposive sampling
   a) Snowball sampling
   b) Quota sampling

Normally used when you are looking for specific/contextual information

1. Convince Sampling

Members of the sample are selected according to convenient accessibility.

Examples:

- Selection of camps which are accessible during the rainy season
- People using a toilet in a refugee camp between 10:00 – 16:00

Can you think of a sanitation related example ?

2. Purposive Sampling

Members of the sample are selected according to the purpose of the study.

Aim of study:
You want to know why 10 tiger worm toilets failed.

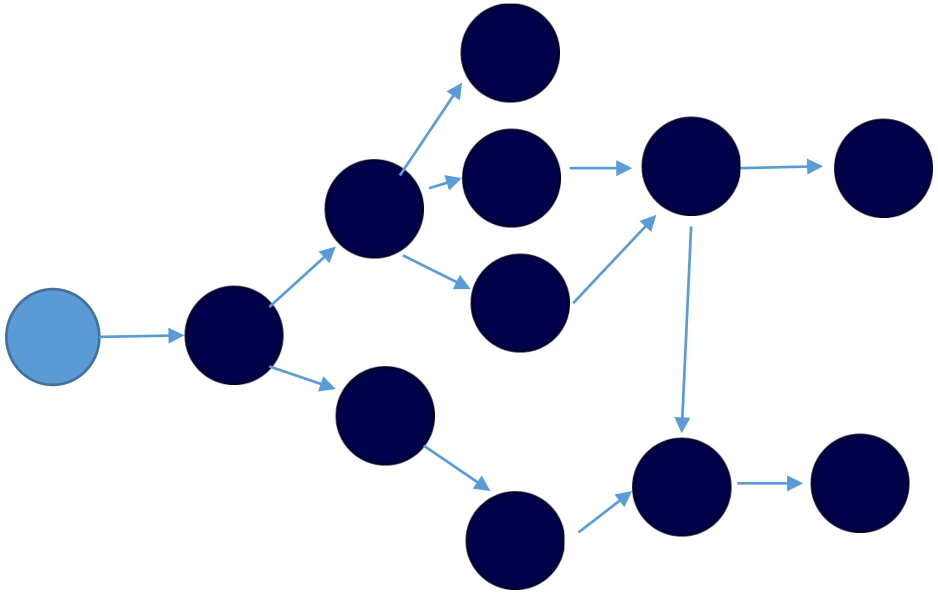
You do structured observations of only these 10 tiger worm toilets

Used: When generalisation is not a concern.

Can you think of other sanitation related example ?

2a. Snowball Sampling

One respondent identifies another respondent.



Used: Difficult to access populations & connected networks

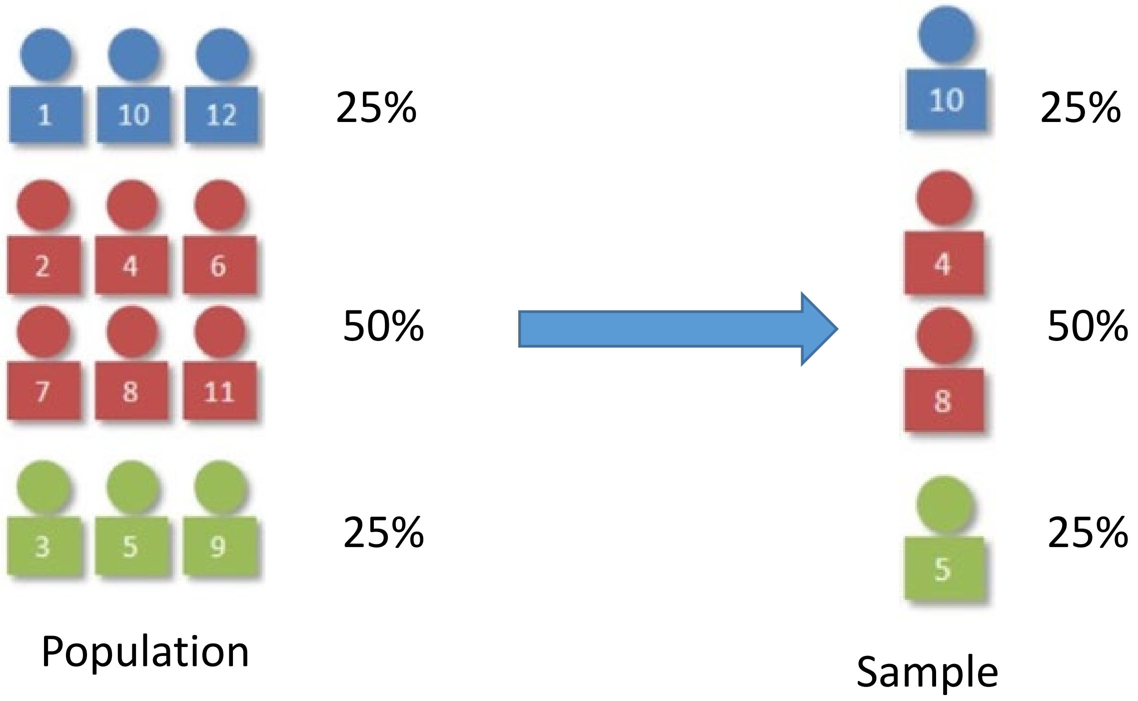
Can you think of a sanitation related example ?

2b. Quota Sampling

Members of the sample are selected according to a certain set of characteristics.

Aim: Explore the sanitation needs in a school.

The school population is 60% male and 40% female. I interview 6 boys and 4 girls.



Can you think of a sanitation related example ?

Samples in Experiments

You may have a sampling strategy, but then what type of sample do you take.

Grab sample: specific time and location (snap shot)

Composite sample: over locations or times

- Time composite
- Effluent collected over 24 hrs
- Space composite
- Core through a latrine

Can you think of other sanitation related example?

You might have more than one sampling strategy…

1. Strategy for selection of sites, communities, households, septic tanks or latrines etc… i.e.

2. Strategy for taking the sample i.e. systematic sample, random or quota

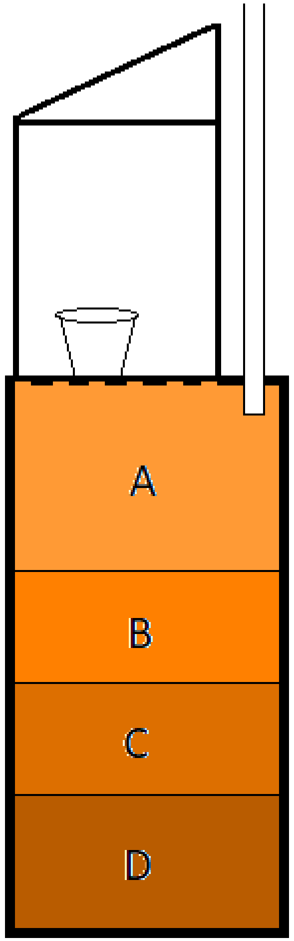
3. Then you may need to describe the type of sample in terms of grab or composite

All need to be related to the purpose of your study or investigation!

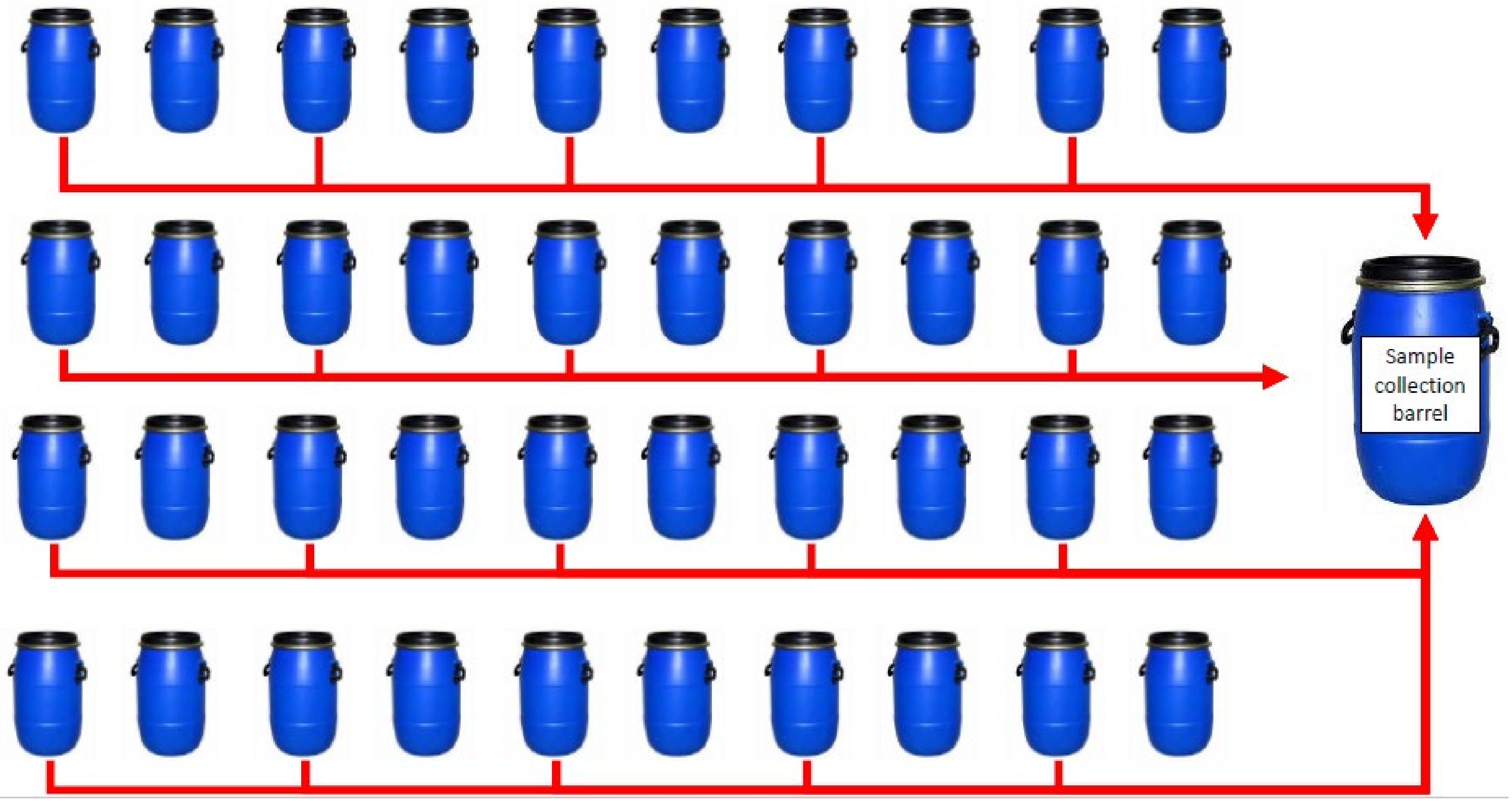
Sampling

How do we sample a pit latrine?

What do you ask yourself first?



Faecal sludge influent & lime treated sludge (1/3)



Sample volume

Is determined by the analytical methods (some methods need higher amount)

Fill sample container completely to avoid contact with air:
- *e.g. determination of dissolved gases*
- *to prevent oxidation*

Fill sample container not completely:

- When sample requires vigorous shaking before taking portions for analysis, e.g. for the determination of suspended solids or bacteria, or with gas producing samples

Collect volume of sample and preserve accordingly to ensure adequate stability of the determinants between sampling and analysis.

Glassware/preservation, according to ISO 5667-3

Determinant	Material of sample container	Method of preservation	Maximum time between sampling and analysis
Ca, Mg, Na, K,..	P, acid-washed	HNO_3, pH 1-2	1 month
Anions (Br, F, Cl, NO_2, NO_3, PO_4, SO_4)	P or G	Cool to 1-5ºC or freeze -20ºC	24 h. respectively 1 month. Filter on-site
Sulfate	P or G	Cool to 1- 5ºC	1 month
Alkalinity, HCO_3	P or G	Cool to 1- 5ºC	24 hours
BOD	P or G	Cool to 1- 5ºC or freeze to -20ºC	24 h. resp. 1 month. in the dark, exclude air
COD	P or G	Cool to 1- 5ºC or freeze to -20ºC	24 h. resp. 6 month. in the dark
Nitrogen, ammonia	P or G	H_2SO_4, pH 1-2 and cooling 1-5ºC	21 days. Filter on-site
Nitrogen, nitrate	P or G	HCl, pH 1-2 or cool to 1-5ºC or freeze –20ºC	7 days resp. 24 h resp. 1 month
Phosphorus, total	P/G/BG acid-washed	H_2SO_4, pH 1-2 or freeze -20ºC	6 months
Phosphorus, dissolved	P/G/BG acid-washed	Cool to 1- 5ºC or freeze to -20ºC	1 month. Filter on-site
Heavy metals, except for mercury	P/BG acid-washed	HNO_3, pH 1-2	6 months
Mercury	BG acid-washed	HNO_3, pH 1-2 , add $K_2Cr_2O_7$ until final conc of 0.05%	1 month

P= polyethylene; G=glass; BG = borosilicate glass

Sample bottles

- components can adsorb to the wall of the container, e.g. benzene to plastic or trace metals to glass

- components can leach from the wall of the container e.g. sodium and silica can leach from glass, organics from plastic

For each determinant is described:

- material of the container: P, G, BG.
- method of preservation: e.g. cooling, acidification, dark/light storage
- max. time between sampling and analysis

On-site measuring during sampling

Measure on-site:

- pH
- EC (electrical conductivity)
- DO (dissolved oxygen)
- Temperature with probes
- Turbidity

The on-site measurement
- Directly in the liquid (or in the container)

Determine before measuring there is:
- complete mixing (settling tank)
- Stratification (layering)
- plug flow (wastewater treatment plant)

Lab & field journal

What should be noted in a Laboratory & field journal?

Making notes

- Make detailed notes
- Make clear notes
- Write down date
- Write down name
- Write clear

In case of sampling:

- Note down name sampler
- Note down location
- Note down time of location

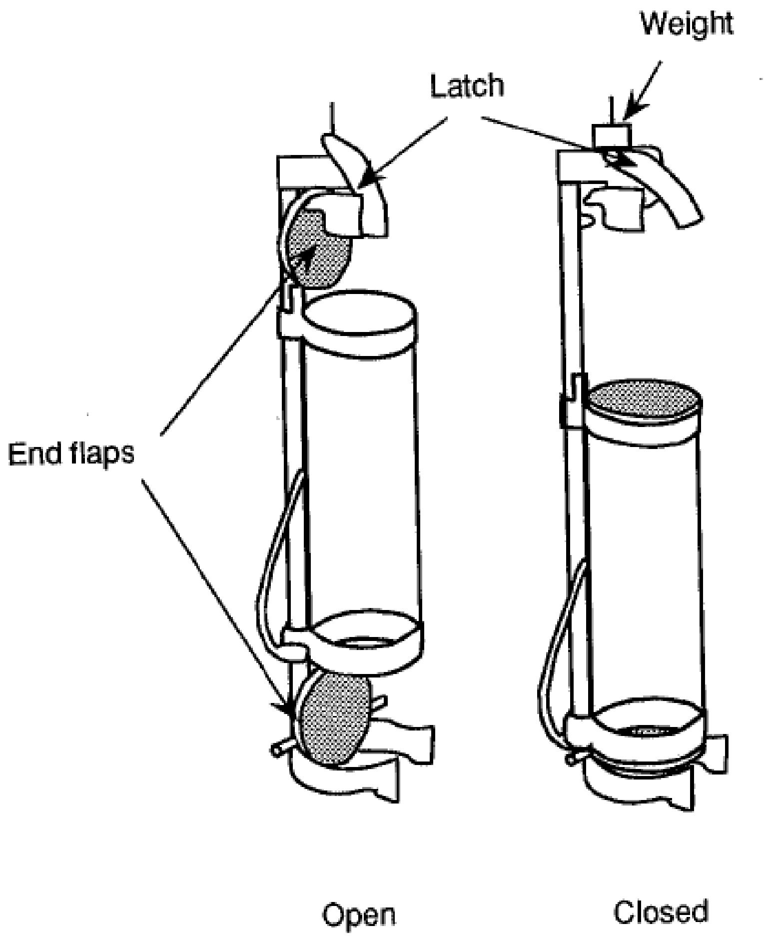
Sampling after/before sampling

- Label the samples (station, date, time, water depth, parameters to be analyzed, preservation chemicals,..)
- Use the correct preservation methods
- Filter, if necessary, over 0.45 µm filter. after that, collecting in clean bottles (to prevent leaching and adsorption from particles)

Ready filter (0.45µm)

BAD labeling

Water sampler



Water sampler (types/brand Ruttner, Nansen, Van Dorn) for taking samples at different depths

When would you use this?

Van Dorn sampler

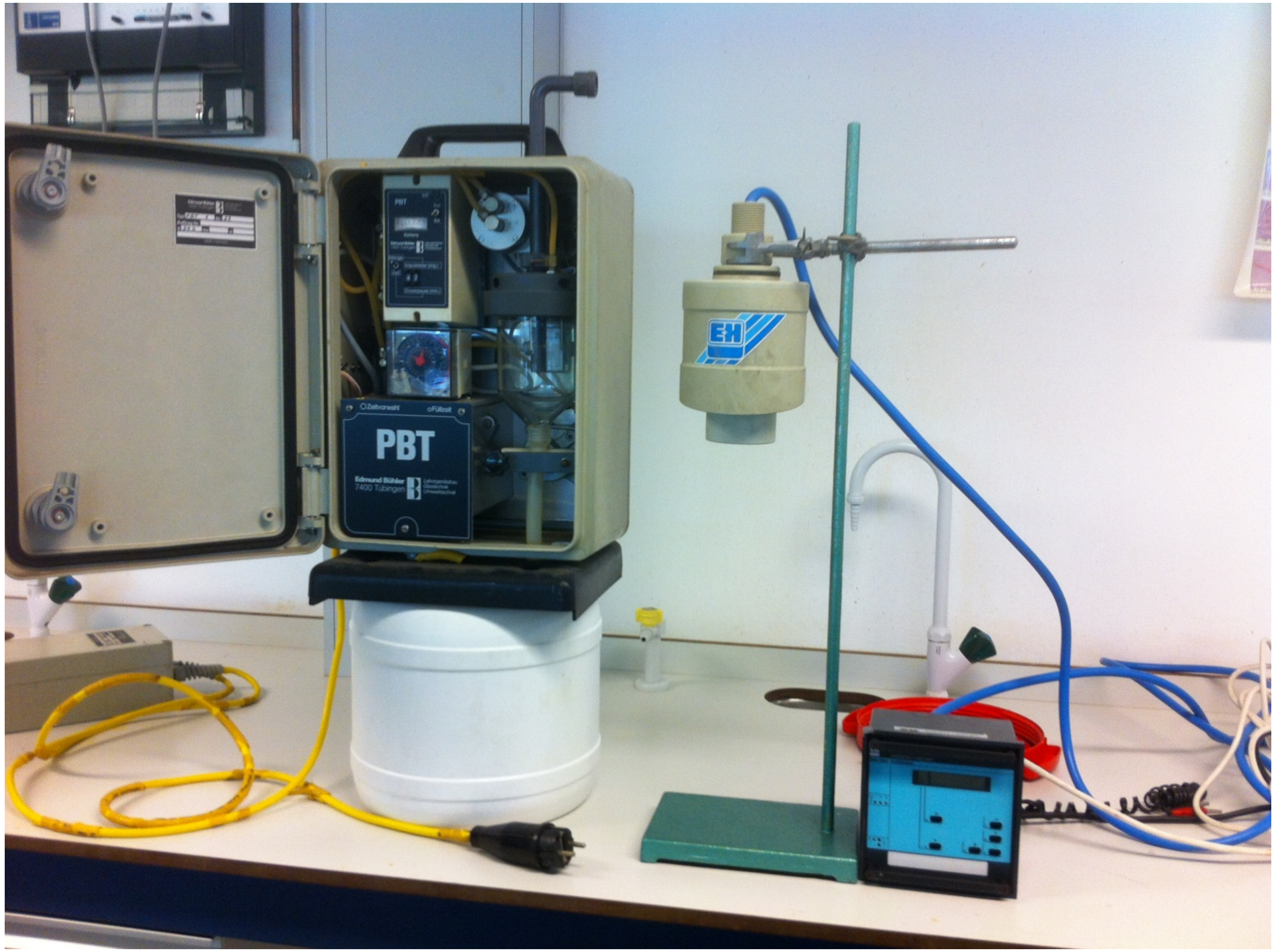
Grab sampler for sediment

Beeker sampler for sediment core samples

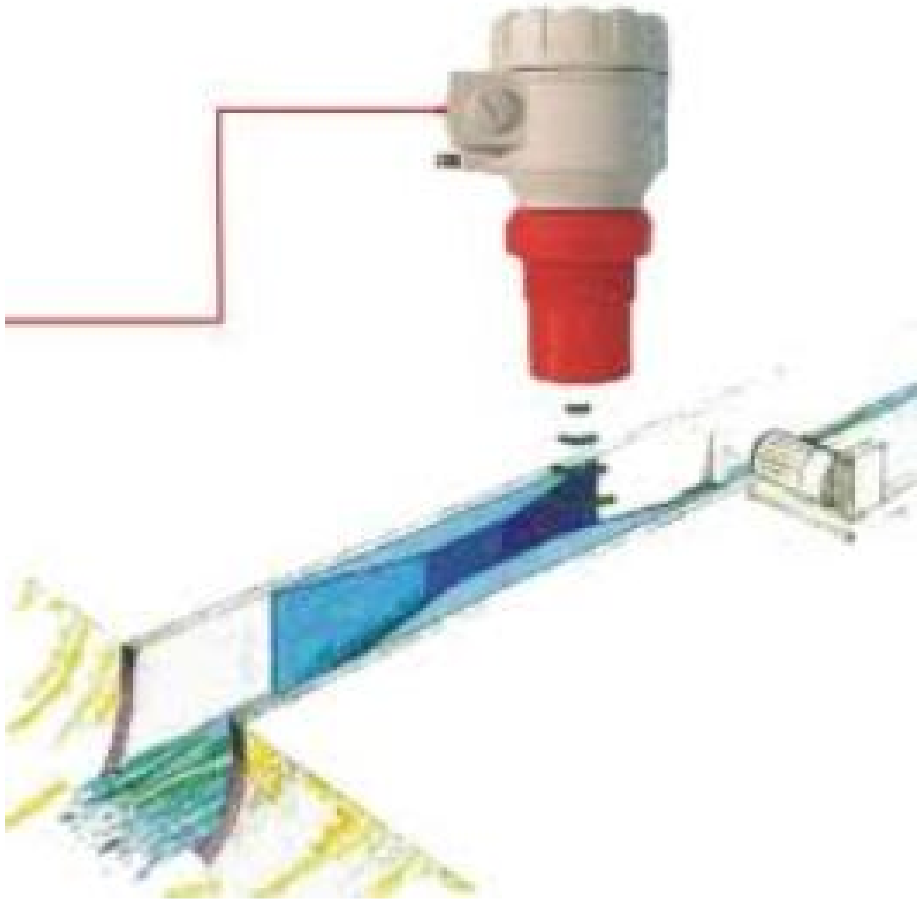
Collection of the core sample

Time integrated sampler

Sampler + ultrasone device for discharge integrated (dependend) sampling

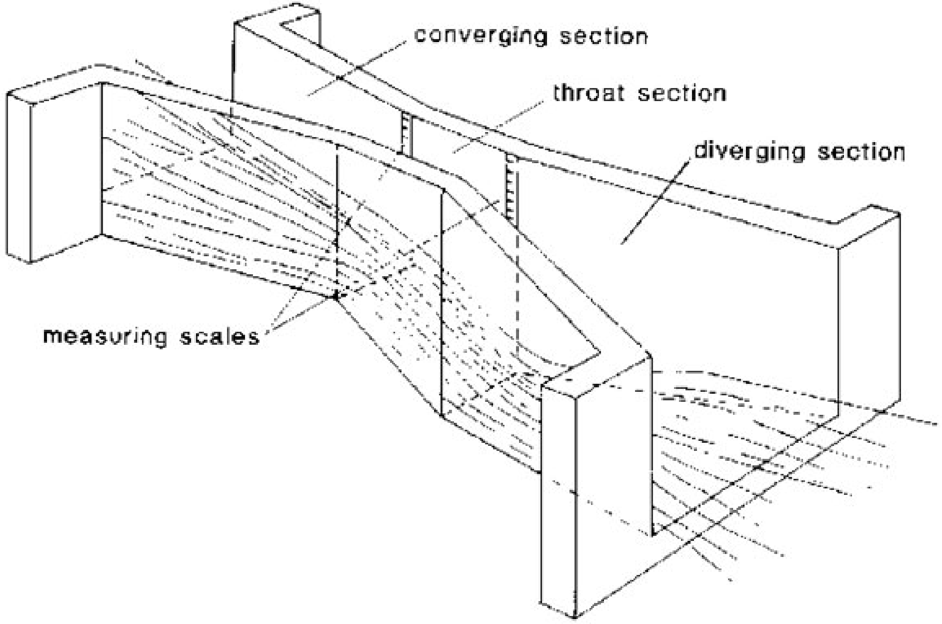


Ulrasone device in combination with flume



Discharge measurement with use of a flume

http://www.v-elin.hr/en/project/flow-measuring-in-open-channels-parshall-flumes/



http://wyomingrenewables.org/renewable-technologies/hydroelectric/wyoming-small-hydropower-handbook/evaluating-resources/measurement-of-head-and-flow/

$$Q = 1.91 \times b \times h^{3/2}$$

Quality Control in the Laboratory

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Quality Assurance vs. Quality Control

Quality Assurance

An overall management plan to guarantee the integrity of data

(The "system")

Quality Control

A series of analytical measurements used to assess the quality (precision and accuracy) of the analytical results

(The "tools")

Definitions (1)

Quality Assurance - QA is defined as the overall program that ensures that the final results reported by the laboratory are correct. So, quality assurance is much more than quality control: it should insure that the right test is carried out on the right sample, and that the right result and right interpretation is delivered to the right person at the right time".

Objective: to prevent mistakes

Quality Control - QC refers to the measures that must be included during each analysis run to verify that the test is working properly. The aim of quality control is simply to ensure that the results generated by the test are correct.

Objective: to correct occuring mistakes

Quality Control

Internal control (daily):

- Blanks, duplicate samples, “spiked” samples.
- Check of ionic charge balance (Electrical Neutrality)
- Compare with standards

Correlation between parameters

- PO_4-P ≦ $P_{tot.}$,
- BOD lower then COD
- NO_3N < TN

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Quality control

Determination through:

- Precision of an analysis

- Coefficient of Variation

- Detection Limit

- Accuracy of analysis

- 95% Confidence Interval

- Control Chart (Shew Chart)

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Quality Control

Precision refers to the repeatability/reproducibility among replicate measurements.

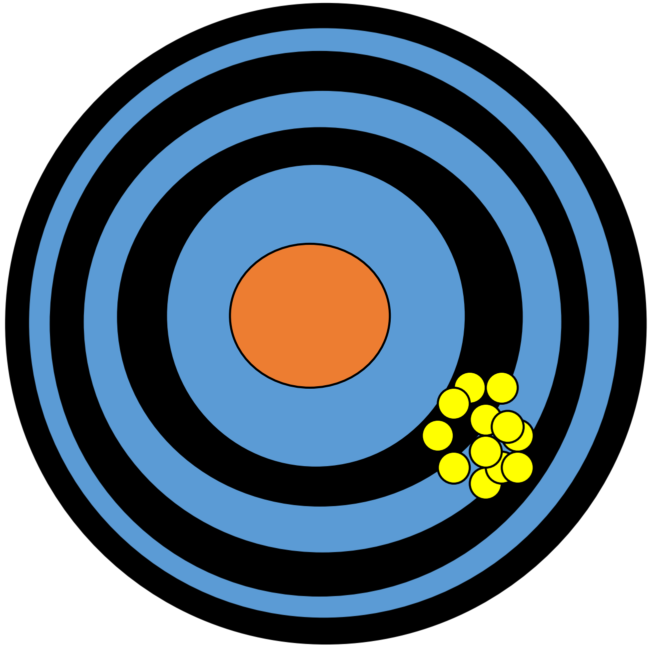
repeatability: the std.dev. calculated from a sample which is measured several times by the same analyst within a short time with the same instruments/ chemicals/ procedures.

reproducibility: the std.dev. calculated from a sample that is measured once by different analysts (with different instruments/chemicals/procedures)

Accuracy refers to the difference between the measured value and known, actual value.

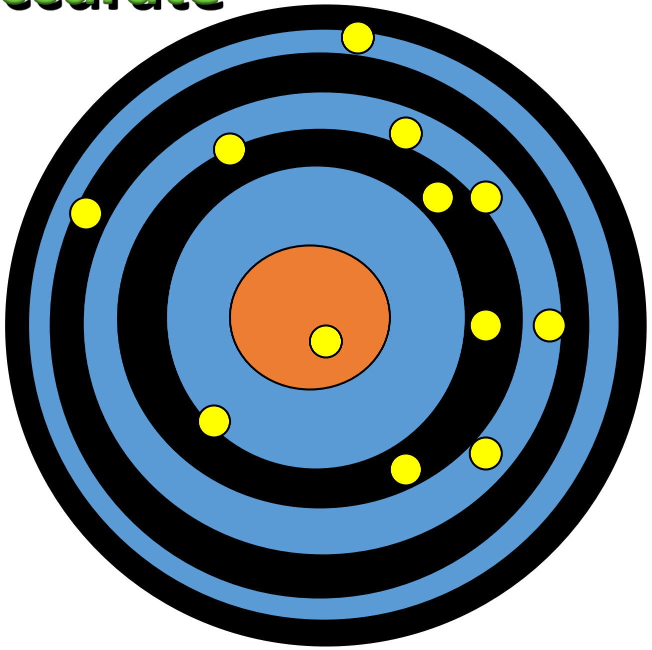
Precision and Accuracy

- **Precise, but not accurate**

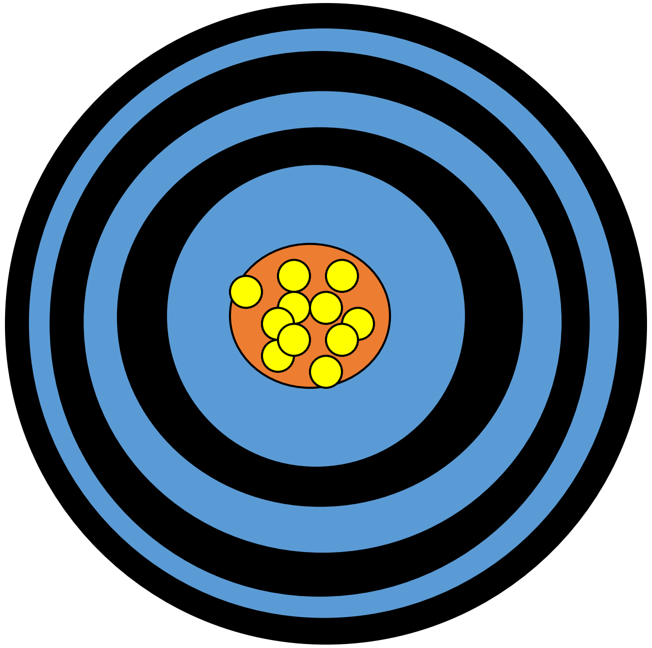


Systematic Error

- **Not precise and not accurate**



Random Error



Precision vs Accuracy

Precision

Expressed as the std dev. at each conc. level

-Use 4 conc. levels
- one near det.limit,
- two intermediate concentrations
- one near the upper limit

-7 replicates at each conc. levels.

Accuracy

Expressed as % recovery

- Known amount of the analyte is added to an actual sample in such a way

that the original concentration is almost doubled (spiking)

- At the 4 conc. levels.

- Express the accuracy for each conc. level as a % recovery of the spiked sample.

Reporting Accuracy

Accuracy is expressed as % recovery:

% Recovery =

$$\frac{\text{(amount found after spiking – initial amount)}}{\text{spiked amount}} \times 100\%$$

QC: Control Charts

Calculate for each sample the mean and the standard deviation.

Construct a control chart (shew chart):

- the mean as its center line
- warning and control lines at $\pm 2s$ and $\pm 3s$ respectively.

The control chart is normally followed over a period of one month.

The analytical procedure is under control when the measured values of the control sample fall within mean$\pm 2s$.

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Standard deviation

Standard Deviation : $$SD = \sqrt{\frac{\sum (x - \overline{x})^2}{(n - 1)}}$$

n = the number of observations (how many numerical values)

Σ = the sum of … in this case, the sum of $(x - \overline{x})^2$

$\overline{x}$ = the mean value

X = the value of each individual observation

The Standard Deviation is an expression of dispersion … the greater the SD, the more spread of the observations …..the lower the precision

Sampling after/before sampling

Standard Deviation (SD)

Is a mathematical expression of the dispersion of a group of data around a mean.

Coefficient of Variation (C.V)

Is the Relative Standard Deviation (also called RSD)

((stdev/average) *100)

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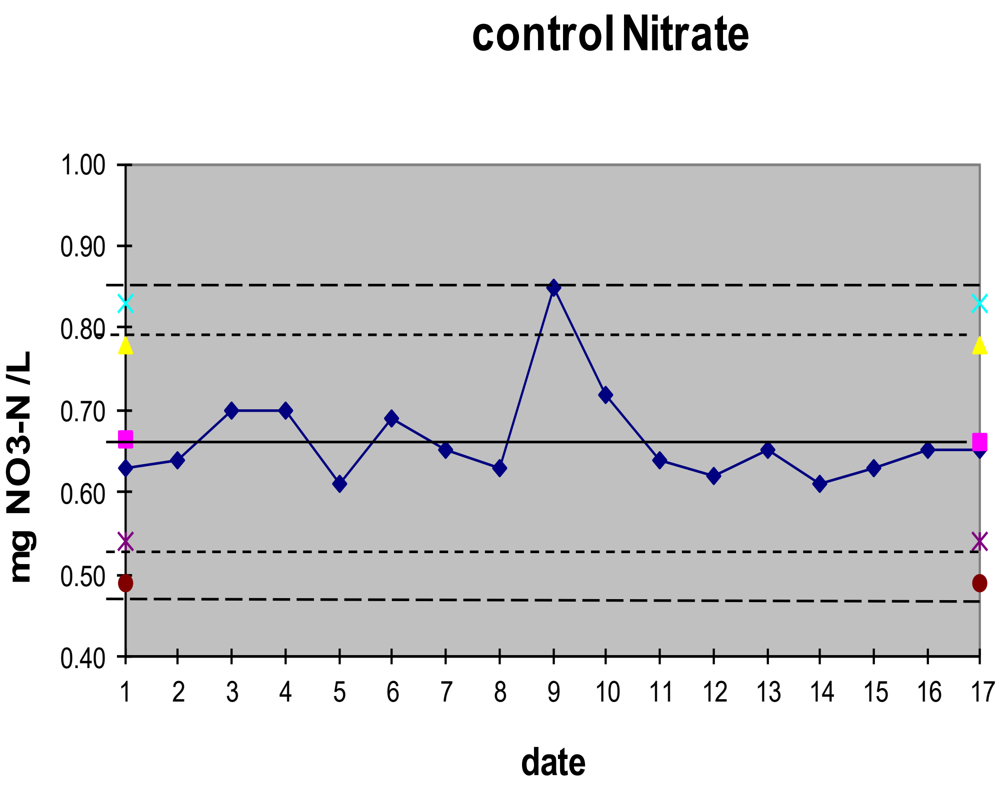
QC: Shew Chart



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Rounding off

<u>If the std.dev is calculated</u>, round the measured values off by using 0.5 * std.dev.

- When the std.dev in this example is 0.0128, results should be presented in 3 decimals because
  0.5 * 0.0128 = 0.0064 (the fluctuation is in the third decimal)

<u>The std.dev itself should be rounded off</u> by using the formulae

- $s/\sqrt{8n}$, here: $0.0128/\sqrt{(8*7)} = 0.0017$

So round off in 3 decimals (the fluctuation is in the third decimal),

here: s = 0.013

Assignment

Measurements:

Sample A : 822.25 g/L

Sample B: 877.32 g/L

Sample C: 850.31 g/L

What is the mean?

What is the standard deviation?

Round off the numbers correctly

Quality Assurance and Quality Control: Summary of important aspects of sampling and analysis

- Sampling program
- Cleaning of materials
- Labels: date/time/locations/remarks
- Calibration of instruments
- Maintenance of instruments and materials
- Use Control Charts

Take home message

- Know about standard deviation
- Know about average
- understand why you sample
- understand how to sample
- Know how to report your collected data