UiO : Kjemisk institutt
Det matematisk-naturvitenskapelige fakultet

Health, Safety and Environment (HSE) regime

At the
Section for Environmental sciences

Safe Job Analysis (SJA)

Risk assessment: CertoClav 12L Autoclave

What can go wrong	Possible Risk	What can be done to prevent accidents	What can be done to reduce the consequences if an accident should occur
Explosion due to very high pressure inside chamber (above 4,1 bars)*	Burns, blindness, and possible risk of death	Work in the hood Wait enough time at the end of the experiment, so that the pressure has fallen to 0 bars and the temperature is below 80°C. Use gloves to take out the samples. Follow carefully the step by step procedure for operating the autoclave (instructions given on autoclave) Always consult an experienced user of the instrument for a demonstration before running the instrument for the first time.	Leave the area if accident should occur & contact safety inspector Vidar Bjekostad, supervisor Rolf Vogt, Alexander Engebretsen or Christian W. Mohr. Place the burned area under cold water. If serious accident should occur contact ambulance and medical help (telephone no.: 113)
Severe burns due to high temperatures (>121°C)	Burns		

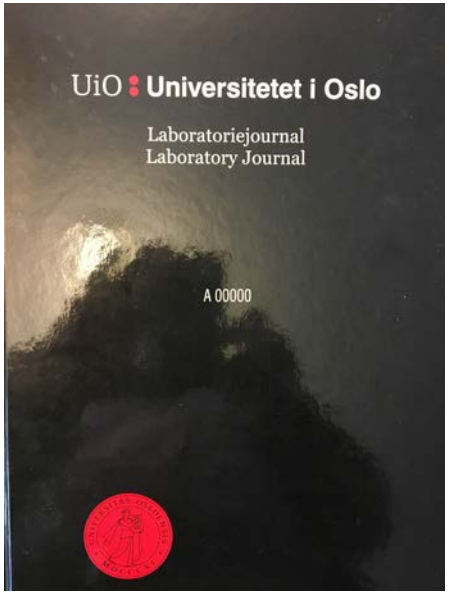
*The autoclave is setup with safety vents in order to avoid explosions. However the instrument must always be treated with the possibility of safety failure.

- Safe job analysis must be made for all analytical operations **representing any danger**
  - What can go **wrong**?
  - What can we do to **avoid** this?
  - What can we do to **minimize** the damage?

- SJA must also be made for the Master project as well as KJM2010 etc.

Resources available on web

- For Environmental analysis students:
  See our internal group pages:
  - https://www.mn.uio.no/kjemi/english/research/groups/environmental-science/environmental-chemistry/internal/
  - If SJA exists then this needs to be read and documented in the Lab protocol notebook and signed by staff (Grethe, Rolf or Cathrine)
    - The Lab protocol notebook is the property of UiO and must be handed in at end of stay
  - If SJA does not exist then one needs to make it and send it to me or Grethe. We will quality control and post it on the internal web



Safety rutines

- In the lab you must use **goggles**
- The doors to chemicals must be **locked**
  - Door to lab must be **locked**
  - Cabinets must be **locked** and key hidden

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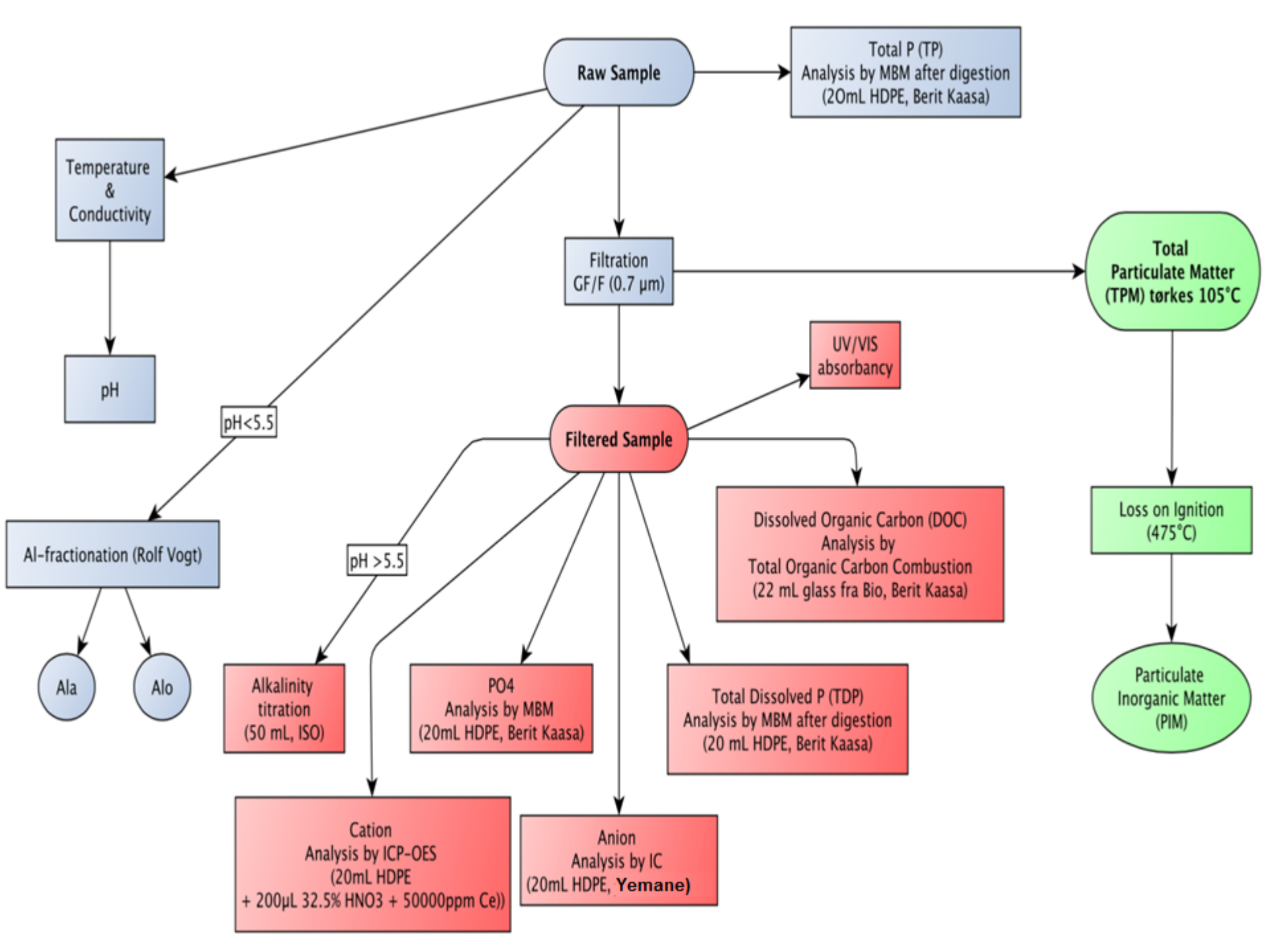
Analysis plan

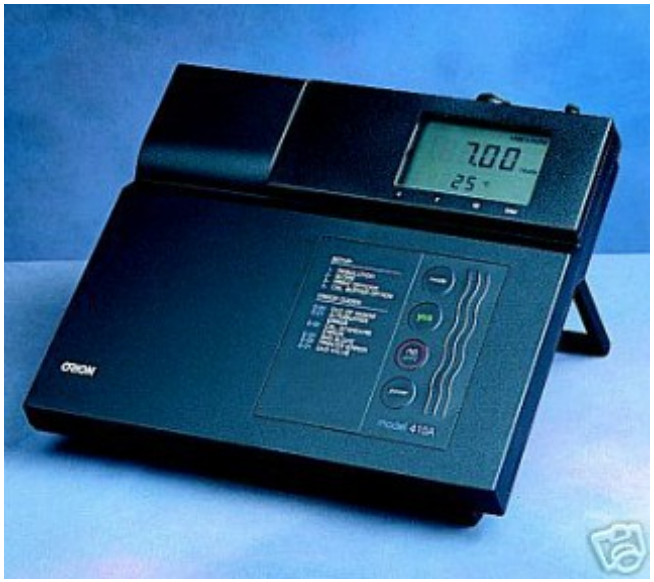
Module 19, KJ;-MENA4020


Raw Sample
Total P (TP)
Analysis by MBM after digestion
(20mL HDPE, Berit Kaasa)
Temperature
&
Conductivity
pH
Filtration
GF/F (0.7 µm)
Total
Particulate Matter
(TPM) tørkes 105°C
UV/VIS
absorbancy
pH<5.5
Filtered Sample
Al-fractionation (Rolf Vogt)
pH >5.5
Dissolved Organic Carbon (DOC)
Analysis by
Total Organic Carbon Combustion
(22 mL glass fra Bio, Berit Kaasa)
Loss on Ignition
(475°C)
Ala
Alo
Alkalinity
titration
(50 mL, ISO)
PO4
Analysis by MBM
(20mL HDPE, Berit Kaasa)
Total Dissolved P (TDP)
Analysis by MBM after digestion
(20 mL HDPE, Berit Kaasa)
Particulate
Inorganic Matter
(PIM)
Cation
Analysis by ICP-OES
(20mL HDPE
+ 200µL 32.5% HNO3 + 50000ppm Ce))
Anion
Analysis by IC
(20mL HDPE, Yemane)

Conductivity

- Conductivity meter
  - The instrument is calibrated using 84 µS/cm calibration solutions
- The measurements are done for **quality control** purposes in order to compare measured and calculated conductivity
- Determined as described in ISO7888 using non-filtered water sample and preferably measured at 25°C.
- The reading is presented as mS m^{-1} (1mS m^{-1} = 10µS/cm)

{H^+} determined using pH electrode

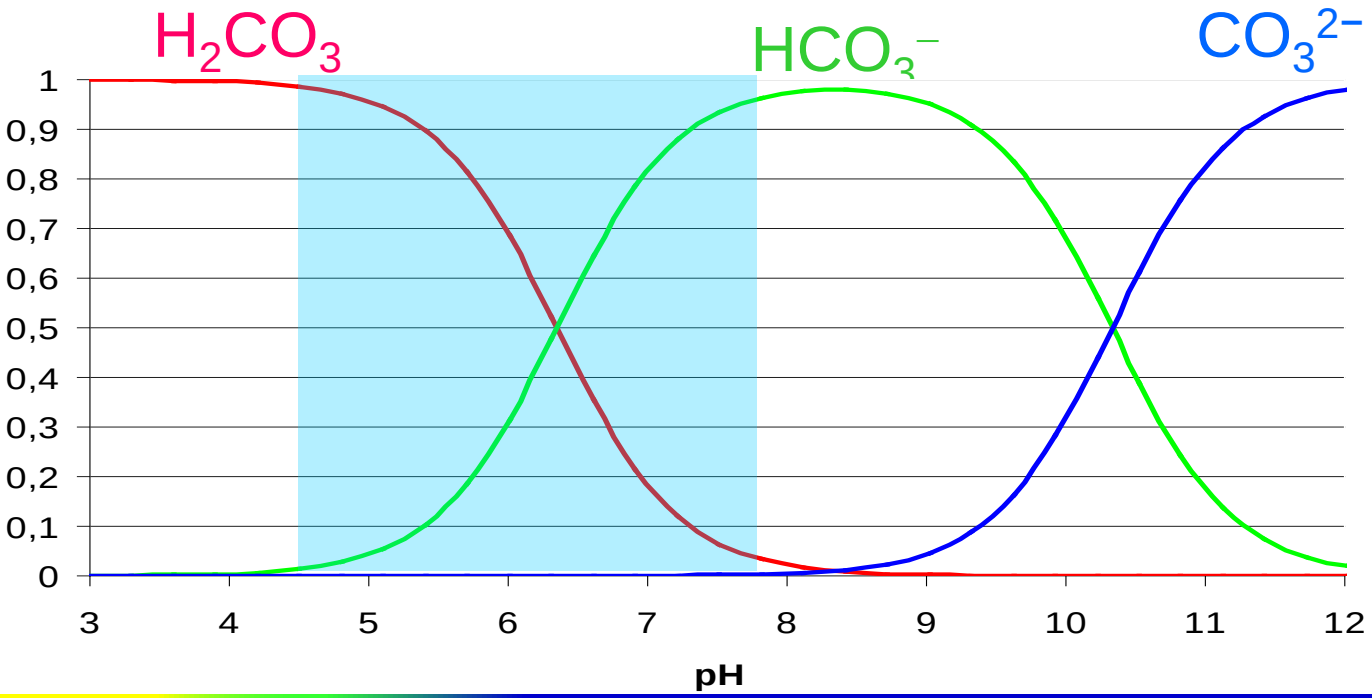


- Orion pH-meter
  with a Ross pH electrode
  - The pH-meter is calibrated with
    pH = 4.00 and 7.01 buffer solutions
- Risk of degassing of CO_2
  - Stir till stable – wait till stable
- Determined as described in ISO10523
  using non-filtered water sample
- Measurements should preferably
  be conducted at 25°C

Total Alkalinity titration

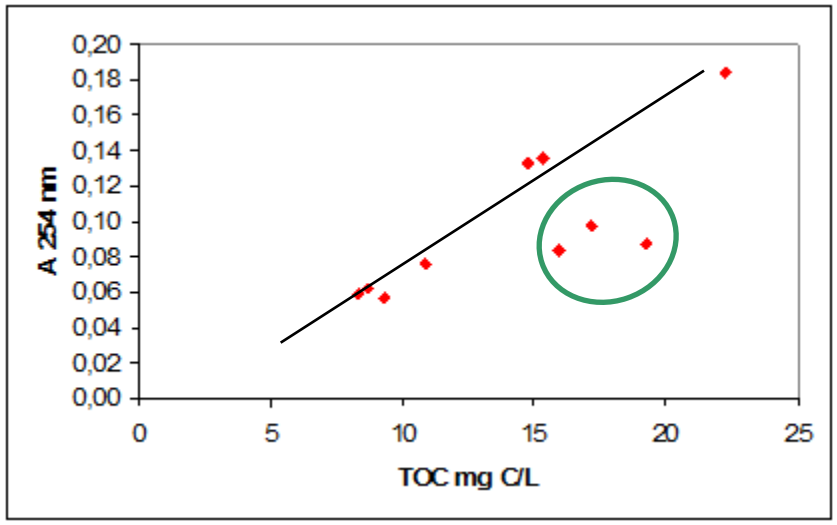
- Determined potentiometrically as described in ISO9963
- Titration with acid to pH 4,5 and preferably measured at 25°C

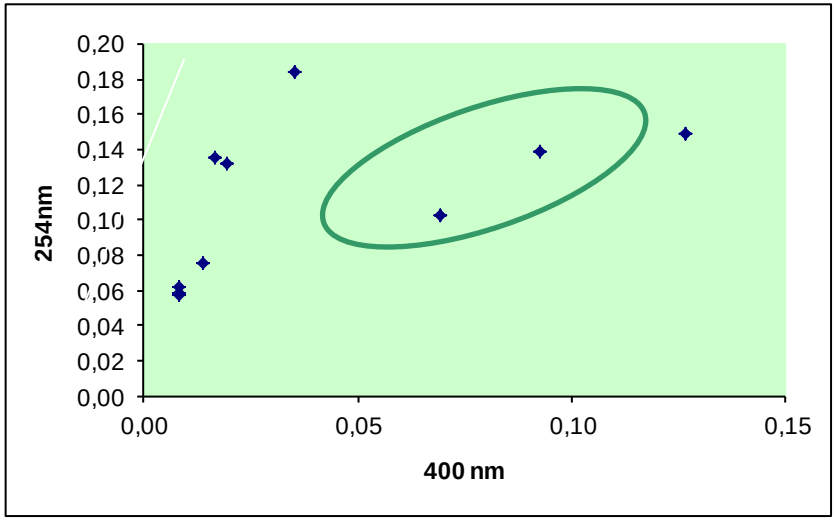
  $CO_3^{2-} + HCO_3^- + 3H_3O^+ \leftrightarrows 2H_2CO_3 + 3H_2O$
- Total alkalinity (=$[HCO_3^-]+2[CO_3^{2-}]$) + H^+ needed to change the pH from sample pH down to pH 4.5 Endpoint in the acid range
- ➔ Also other weak acids (A^-, $Al(OH)_n^{3-n}$)



UV/VIS MAS

- Measurement of absorbency @ λ254, 400 and 600nm
- Absorbency at UV 254 nm is commonly used as a proxy for Dissolved Natural Organic Matter (DNOM)
  - Strongly correlated with TOC due to conjugated double bonds chromophores
- Algae adsorb light at UV 254 nm, but more than DNOM at VIS 400 nm





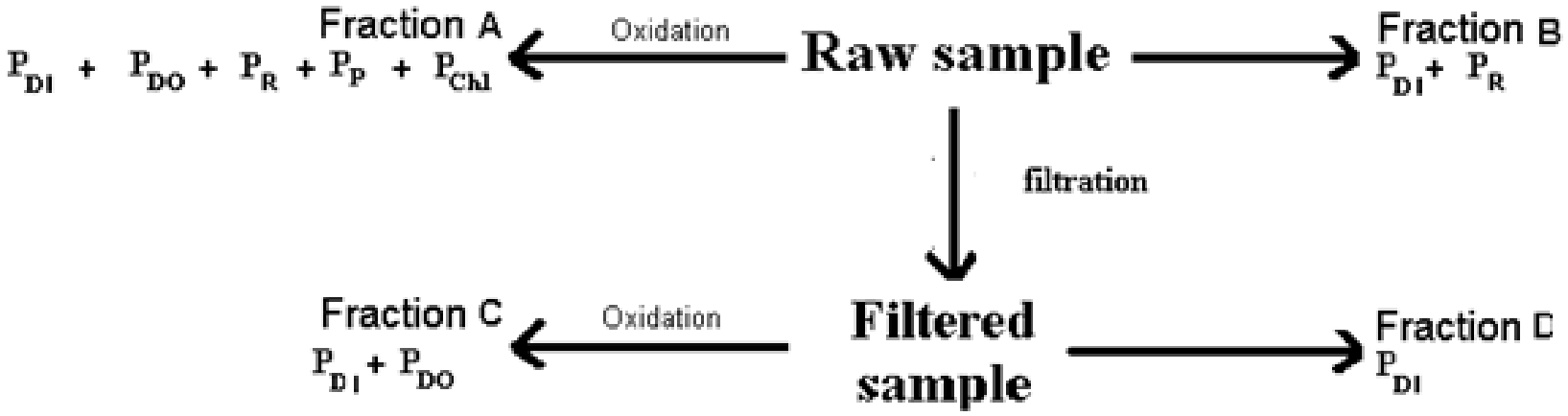
P determination

- Orthophosphate reacts with ammoniummolybdate to a yellow-coloured phosphorousmolybdate acid, that is reduced with ascorbic acid in the presence of antimony to a strongly blue coloured complex
- This colour is measured photometrically as described in Norwegian Standard (NS 4724)

- At higher concentrations (soil extracts) P conc. is determined by ICP-OES
  - Detects higher cons. then the colorimetric method due to inclusion of non-labile species (org.-P etc)

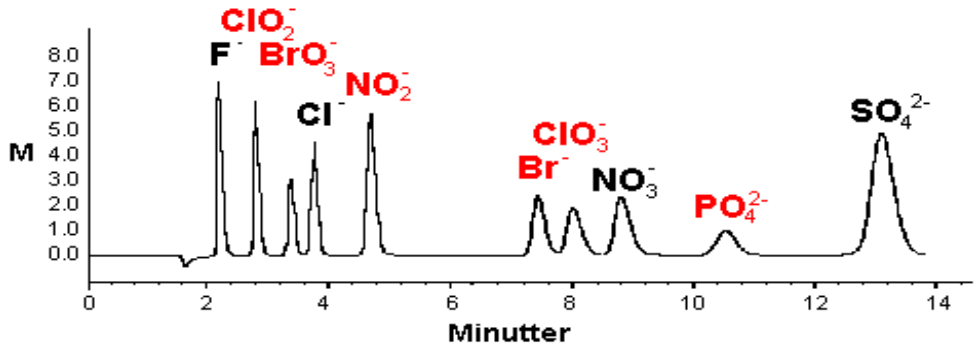
Nutrient fractionation

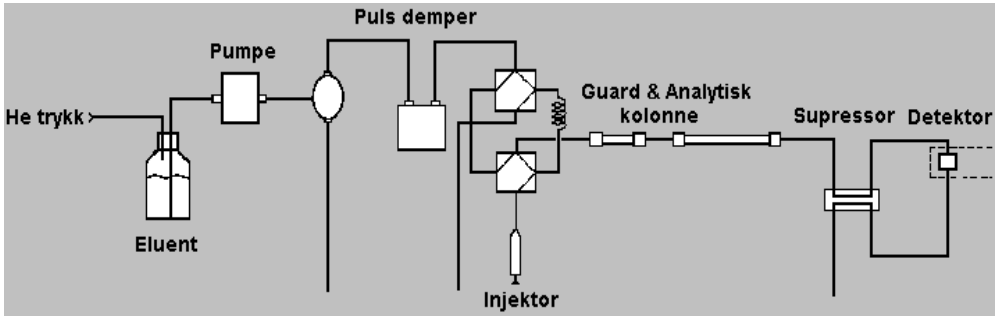
P_{DI} = Orthophosphate = Fraction D
P_{DO} = Dissolved organic P = Fraction C - D
P_R = Reactive pool of P = Fraction B - D
P_P = Particulate P = Fraction A - C



Major anions to be determined by Ion Chromatograph (IC)

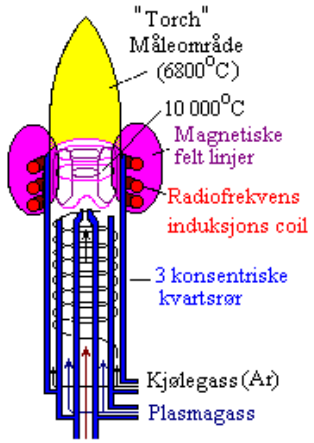
- Principle
  - The sample is injected in a flow of eluent
  - The analyte ions are separated by different degree of binding to the active sites on the ion exchange material
  - Cations are exchanged with H^+
  - The activity x specific conductivity of the analyte along with H^+ in the eluent stream are measured by means of a conductometer

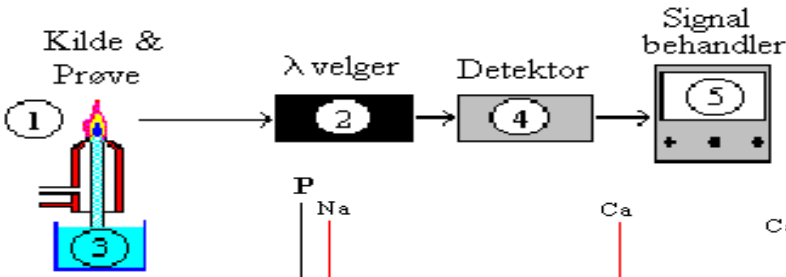


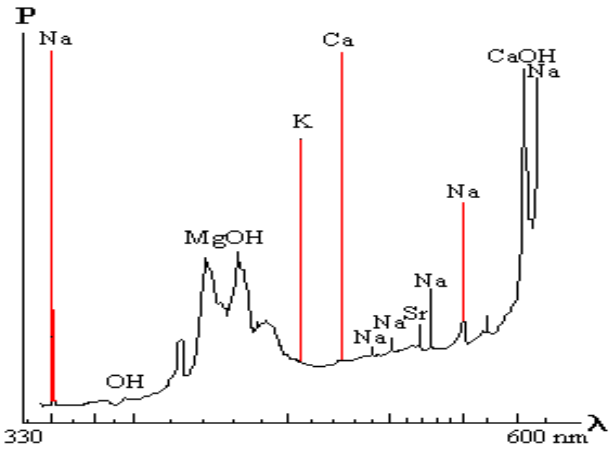


Major cations to be determined by ICP-AES/OES or MS

- Ohmic heating
  - The heat arises from interaction between the ions and electrons and a fluctuating magnetic field which causes the charged particles to move in circular orbits







- Atomization: Plasma
- Detection technique: Atomic/Optic **emission** spectroscopy; AES

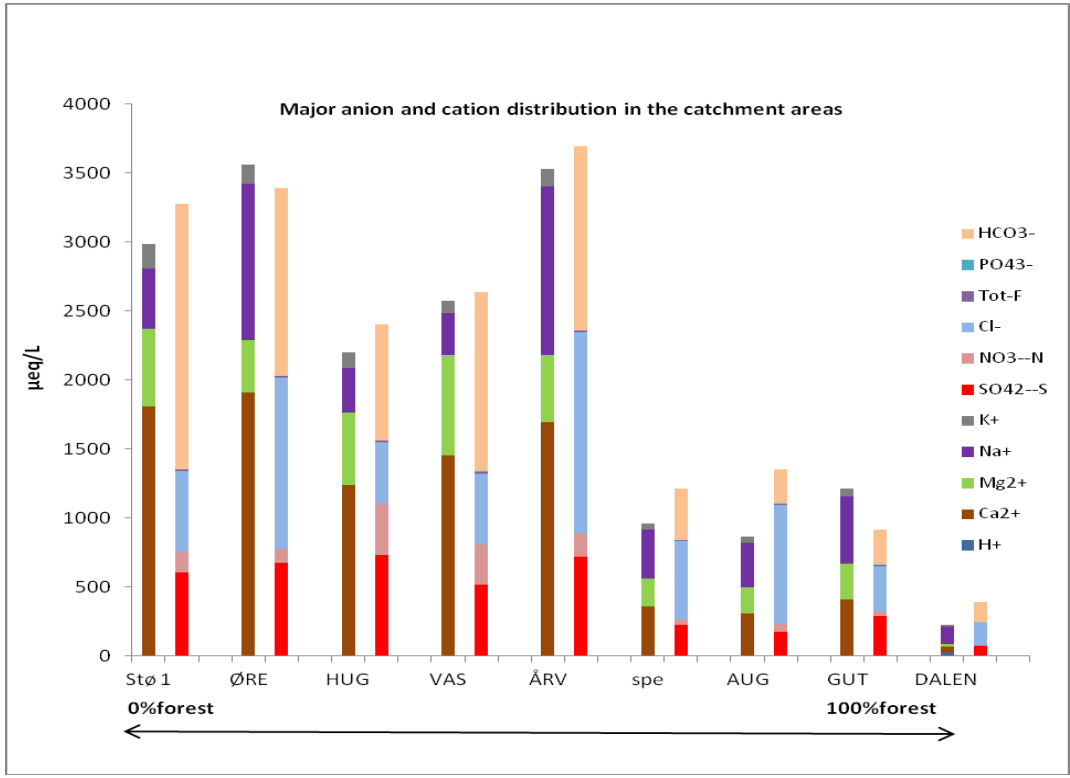
Total organic carbon

- Analytical chemistry lab Ø 104
- High temperature (680ºC) catalytic combustion analysis on a Shimadzu TOC-5000A instrument
- Principle:
  - The organic carbon is combusted to **CO_2** by high temperature and catalysis.
    The amount of CO_2 produced is measured using an **IR detector**

- Analytes measured may include: TC, IC, TOC, NPOC, and POC

Problems with analysis of major anions and cations in water

- Anions are analysed in raw water
- Cations are analysed in water digested in acid (HNO_3)
  - Colloidal material (<0,45µm) is included in the cation analysis but not in the anion analysis



QC of data

- After the analysis the data must be compiled and quality controlled by
  - ion balance and
  - agreement between measured and calculated conductivity
- For this purpose you may use the Data compilation and QC worksheet available at http://folk.uio.no/rvogt/KJM_MEF_4010/