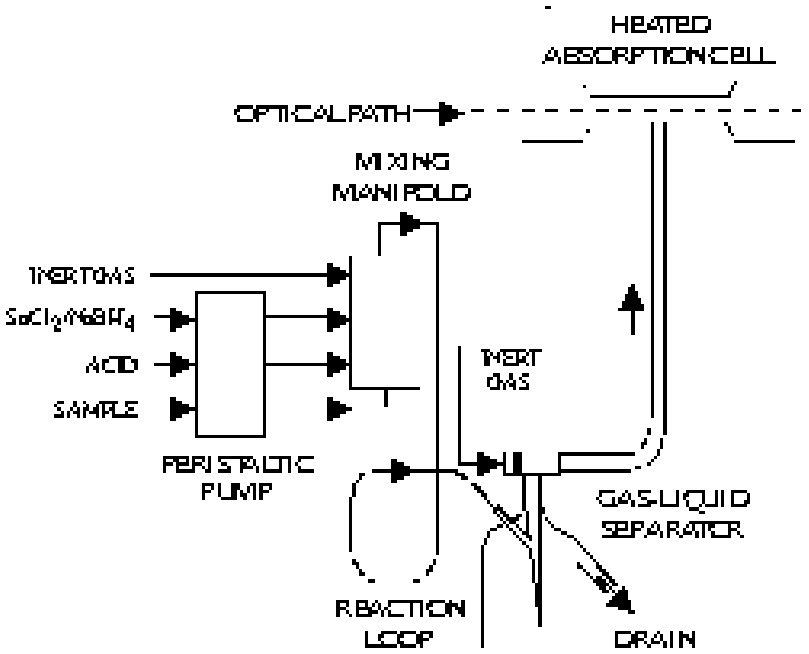
Functional Diagram of VGA



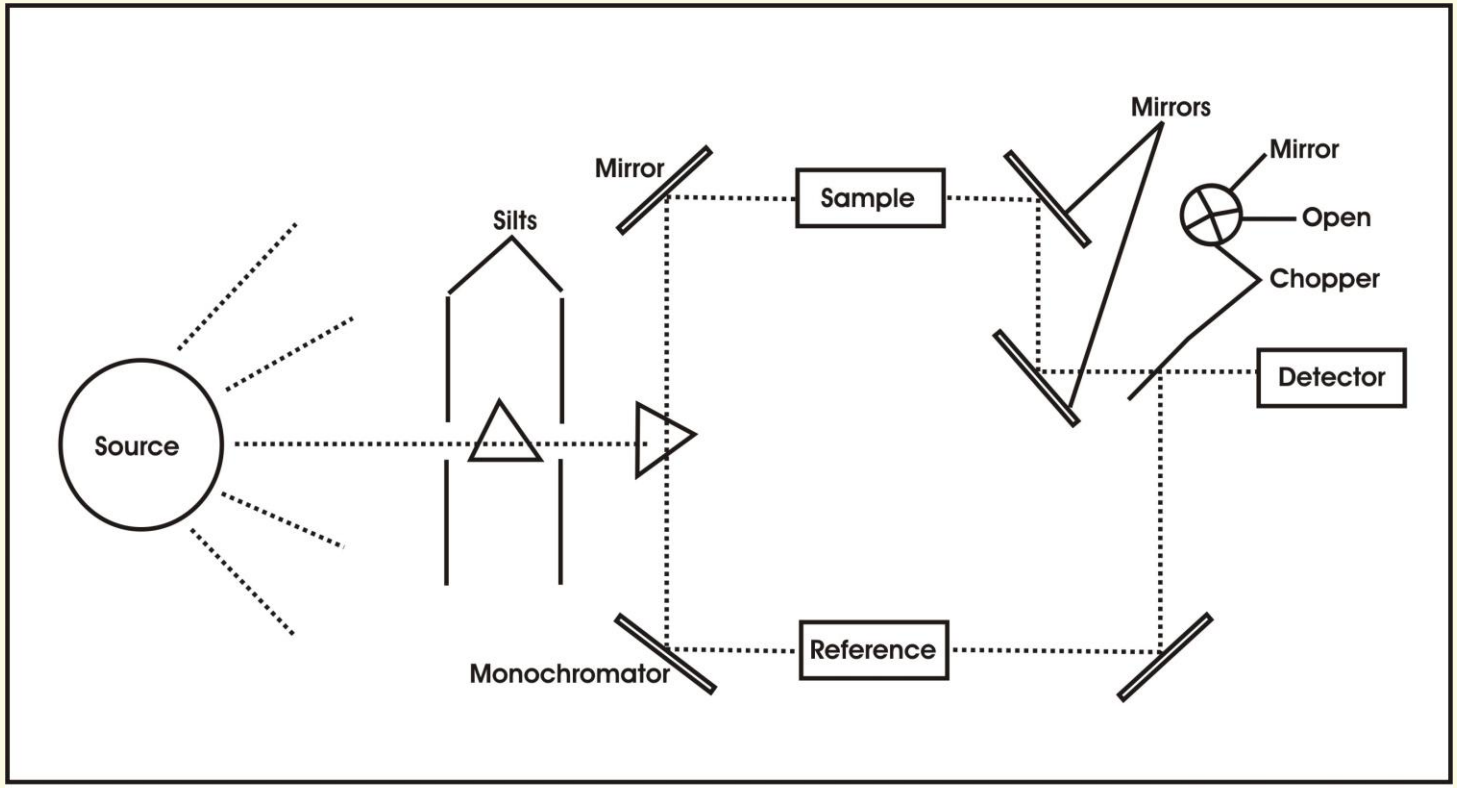
GOOD LABORATARY PRACTICE AND SAFETY

- Use only INSTRUMENT GRADE Acetylene (99.5% or better).
- Acetylene cylinders must not be used at pressures below 500 kPa. Doing so may allow acetone to enter the fuel line and damage the instrument.
- Do NOT use oxygen or oxygen-enriched air.
- Heat, vapours and fumes generated by the flame can be toxic, hazardous to your health.
- Switch on the exhaust before lighting the flame.
- NEVER use glass bottles for waste. Use only plastic beakers or containers.
- Never aspirate organics with a density <0.75 g/mL as a flashback may result.
- Never bring acetylene in direct contact with Cu, Ag, Hg, Cl or grease as an explosion might result.

GOOD LABORATARY PRACTICE AND SAFETY

- **Compressed gases should always be handled with care and in strict accordance with the gas manufacturers instructions.**
- **Keep gas cylinders cool and secured to the well in an upright position with a thick chain.**
- **The acetylene line pressure must not exceed 100 kPa as the gas may spontaneously explode.**
- **If gases are allowed to escape into the laboratory, an explosion or suffocation may occur. Check all gas lines for gas leaks, and ensure proper ventilation in the laboratory.**
- **Do not use a flame for gas leak testing but use a weak soap solution such as "Snoop".**
- **Never pass acetylene through copper, brass or tubing where there is a greater than 65% copper as an explosion may result.**

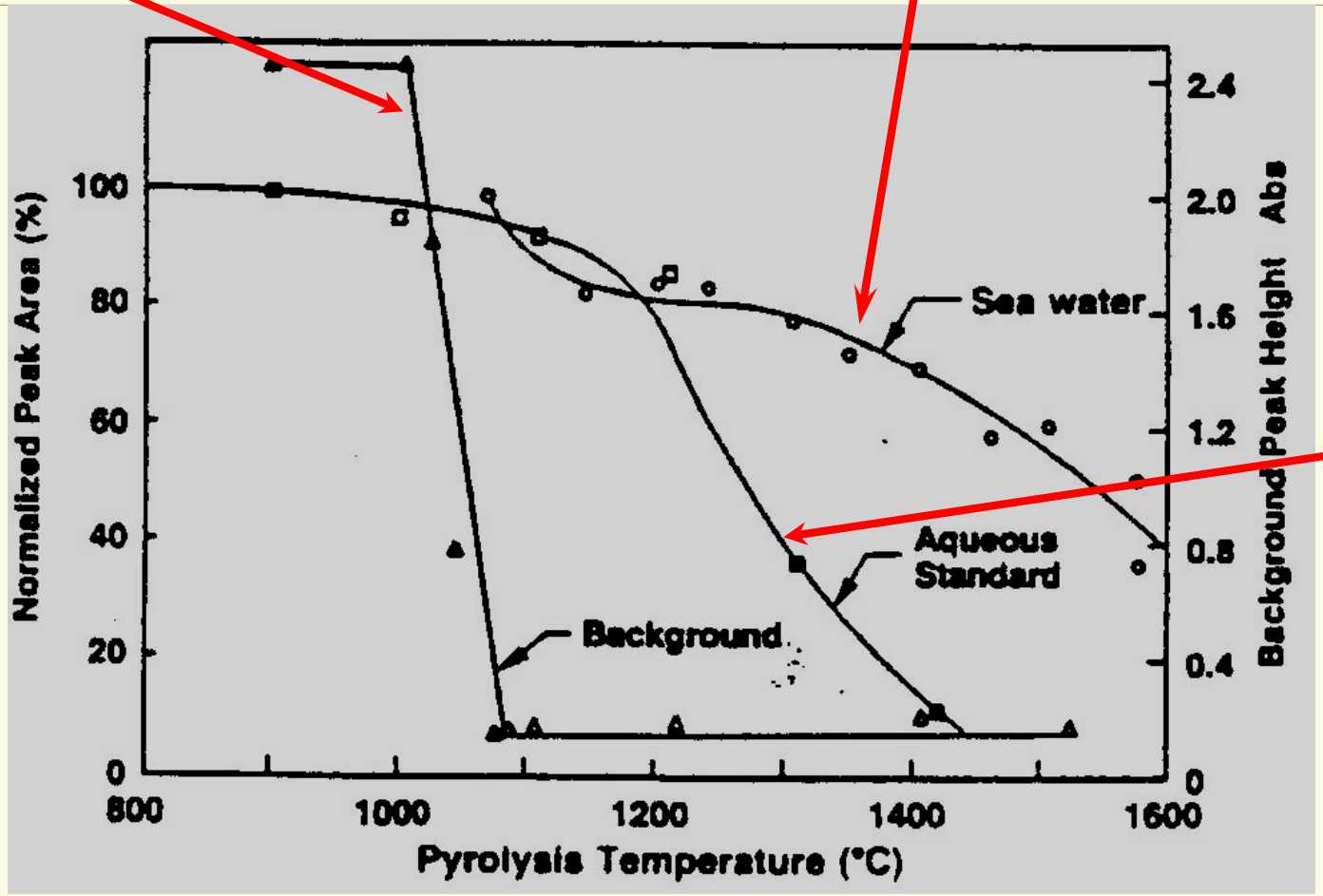
Schematic diagram of double beam Spectrophotometer



Manganese in Sea Water

Excessive Background signal from NaCL

Less Manganese lost over 1050^0C



Large loss of analyte

Interferences

- separates the analyte element from the chemical matrix
  - eliminates matrix interference effects
  - minimises background absorption
- improves efficiency of atomisation for certain elements over conventional FAAS
  - ppm to ppb/ppt levels of detection
- hydride forming elements
  - As, Bi, Pb, Sb, Se, Sn & Te
- mercury
  - CVAAS / amalgamation

Interferences

Spectral

very rare in the gas phase

Kinetic

- caused by varying rates of development / liberation of the hydride
- incomplete digestion of samples - high organic content
- foam production can retard hydride production
- add anti-foaming agent
- n-octanol / Dow Corning AntifoamB®
- peak area integration eliminates most effects

Interferences

Oxidation state

- only AAS technique which is oxidation state sensitive
- higher sensitivity is obtained for the lower oxidation state

Group 5A elements - As, Sb & Bi

- +3 oxidation state
- +5 oxidation state (<2x signal decrease)

Group 6A elements - Se & Te

- +4 oxidation state only gives signal

Pre-reduction necessary

- Group 5A elements - KI/Ascorbic acid or KI/strong acid
- Group 6A elements - hot 4-6M HCl
- treat standards as samples

Interferences

Chemical

- most mineral acids at high concentration will only slightly depress signal
- HCl, HNO_3 & H_2SO_4 used for sample/standard preparation
- HF & $HClO_4$ remaining from digestion must be diluted
- peak area integration can eliminate most effects
- major interferences from Group 8 elements (Fe, Co, Ni, Ru, Rh, Pd, Os, Ir, Pt) or Group 1B elements (Cu, Ag & Au)
- increase acid concentration
- add complexing agent
- add masking agents (Fe in presence of Ni)
- well documented

Interferences

Gas phase

- mutual interaction between analyte & competing element
- ‘competing’ for hydrogen free radicals
- hydrides with other hydrides
- depends on speed of hydrides reacting
- add KI
- add ions that interfere with interferent - Cu(II) with Se/As
- well documented

Error

Systematic

- Hg contamination - soak/clean all glassware & tubing with HNO_3 + water
- Hg adsorption (to surface of vessels) minimised by acidifying to pH2.5
- purity of reagents & acids
- volatilisation during sample preparation
  - closed vessel digestion

Amalgamation Technique

- Slowly liberate Hg vapour from system
- Hg is collected by amalgam formation on gold ribbon in system
- gold ribbon rapidly heated to 500-700^{o}C
- concentrated Hg vapour released gives higher signal
- ppt levels of detection possible

inject sample
smoothly dry sample
ash / char sample to remove sample matrix
efficiently atomise analyte
clean tube before next sample injection

sample volume deposited on base of tube

injection depth

- volume dependant
- 10-40 μL
- sample dependant

rate of injection

observe using dental mirror

dry temperature based on solvent
volume dependant
initial dry step just less than b.p of sample
second dry step just above b.p of sample
slow ramp used between steps
max inert gas flow used (3L/min)
observe using dental mirror

rapidly generate as many free ground state atoms as pos
GBC recommend atomise temperatures - methods manu
atomise hold time will vary with analyte - 1 to 2s ‘read’
rapid atomise heating rate - 1000^{o}C to 2000^{o}C/s
ensure whole signal is captured
inert gas stop used (0L/min)
2s gas stop recommended before atomise step

rapidly generate as many free ground state atoms as pos
GBC recommend atomise temperatures - methods manu
atomise hold time will vary with analyte - 1 to 2s ‘read’
rapid atomise heating rate - 1000^{o}C to 2000^{o}C/s
ensure whole signal is captured
inert gas stop used (0L/min)
2s gas stop recommended before atomise step