Flow Injection Analysis (Chapter 33)

Automatic - no feedback to control

Automated - feedback to control

Advantages of Automation:

(1) Economic

operator costs

reduced waste

human errors

(2) Speed

(3) Precision

Disadvantages of Automation:

(1) Cost

Initial purchase

Setup cost

(2) Inflexible

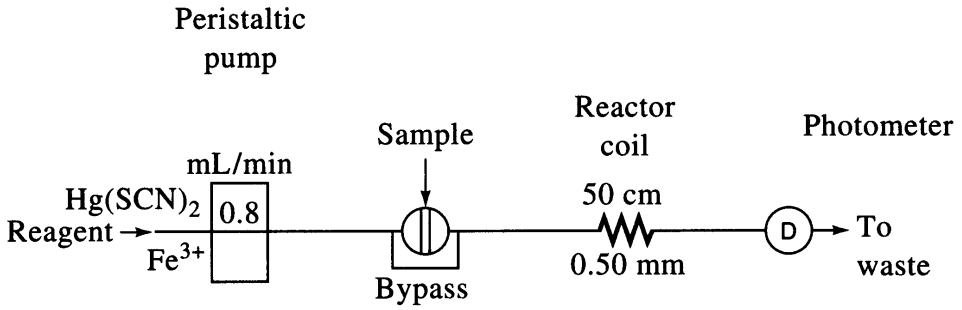
Two types of auto analysis

(A) Discrete/batch - sample remains isolated throughout (robot, no cross-contamination problem)

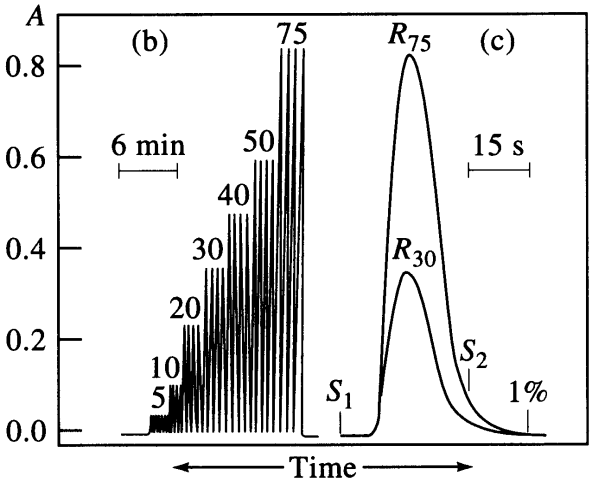
(B) Continuous - sample introduced into stream (simpler instrumentation, more efficient)

Flow Injection Analysis (FIA) developed in response to need for automated analysis - especially in clinical field

Fig 33-1



(a)



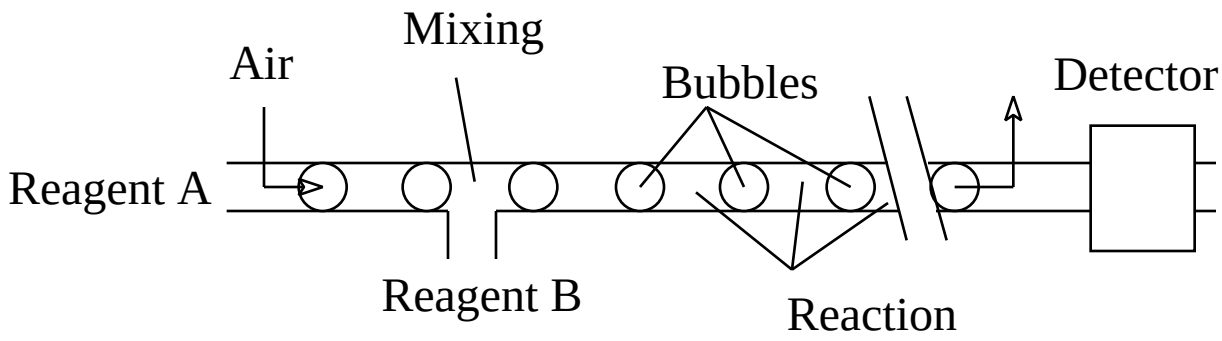
(b)

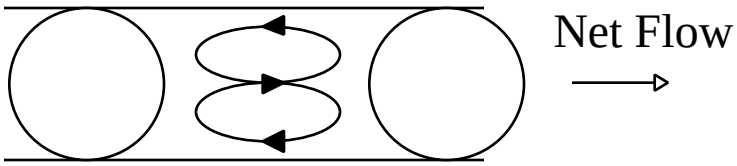
In first instruments, regular air bubbles introduced to

(i) reduce dispersion (diffusion along tube)

(ii) improve mixing

Air-segmented continuous flow analysis (ASCFA)





Bolus Flow Profile

But, bubbles

(i) increase analysis time

(ii) require bubble introduction/removal (bubble gate)

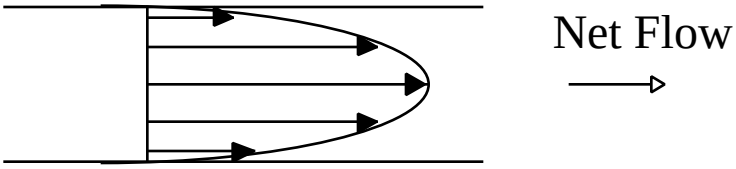
(iii) increase startup/settle time

However, in FIA system with narrow id tubing and low flow rates no need for bubbles

At injection, sample zone (plug) concentration profile is rectangular

Profile changes downstream

(i) Laminar flow profile develops due to friction with walls



Laminar Flow Profile

(ii) Radial diffusion from center to walls of tube

(iii) Longitudinal diffusion in forward and backward flow directions

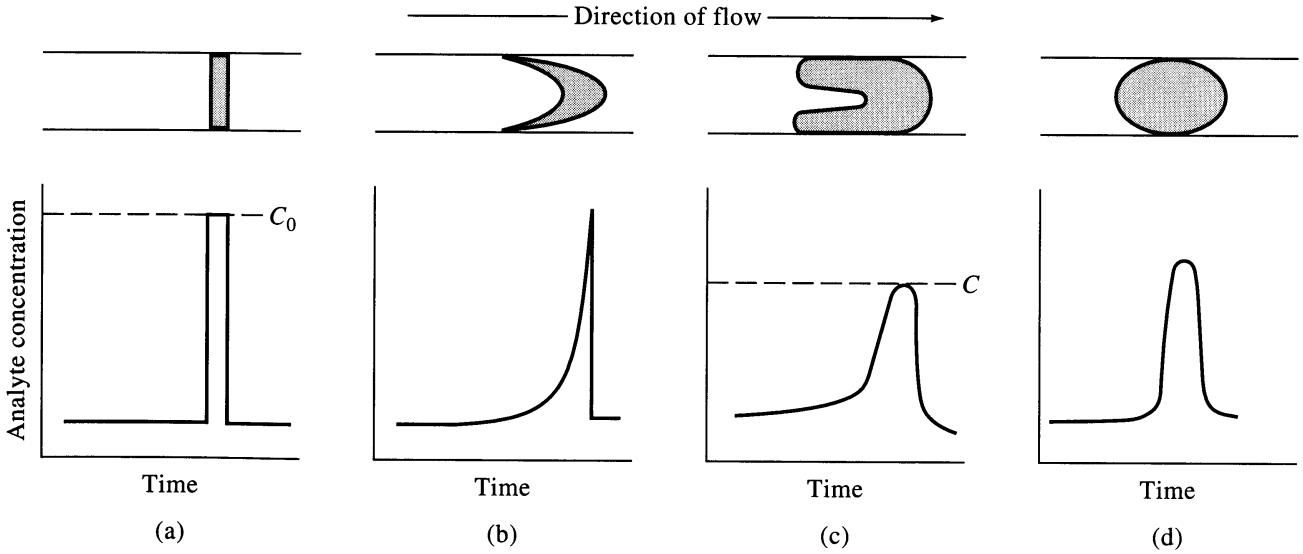


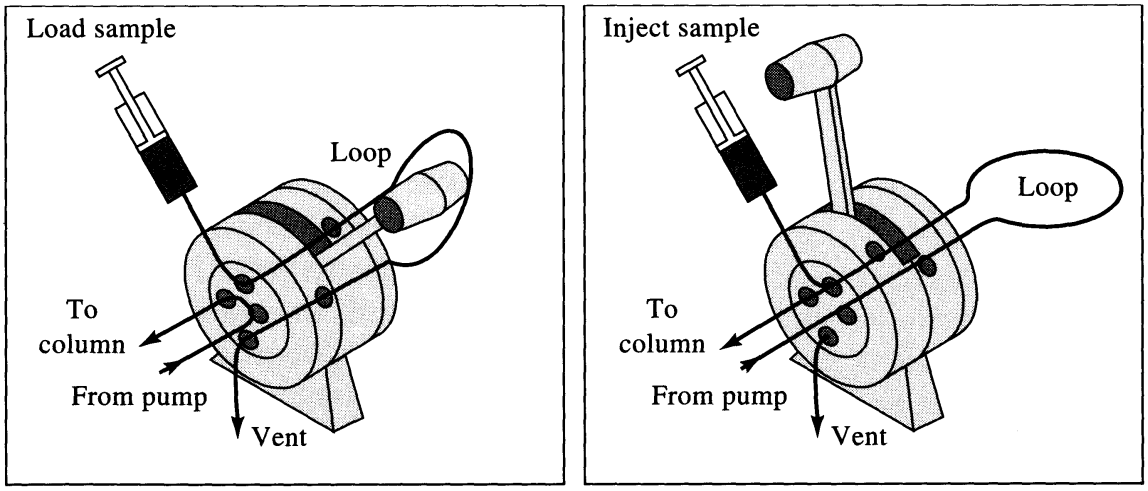
Fig 33-5

When flow rate/tube id is small (d) is approached - no need for bubbles!

Radial diffusion from walls to center helps minimize cross-contamination between sample plugs

Sample Introduction

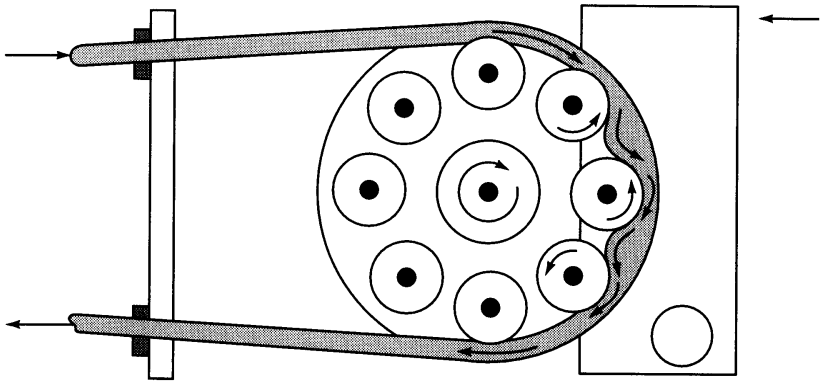
Injection valve: (Fig 28-7)



(i) allows loading of specified volume of analyte (5-200 µL)

(ii) rapid injection of sample plug into flow without disturbing tube flow

Pumps: (Fig 33-2)



(i) Variable speed peristaltic pump

(ii) Flow rate (0.0005 to 40 mL/min) controlled by pump speed and tube id

Tubes and Reactors:

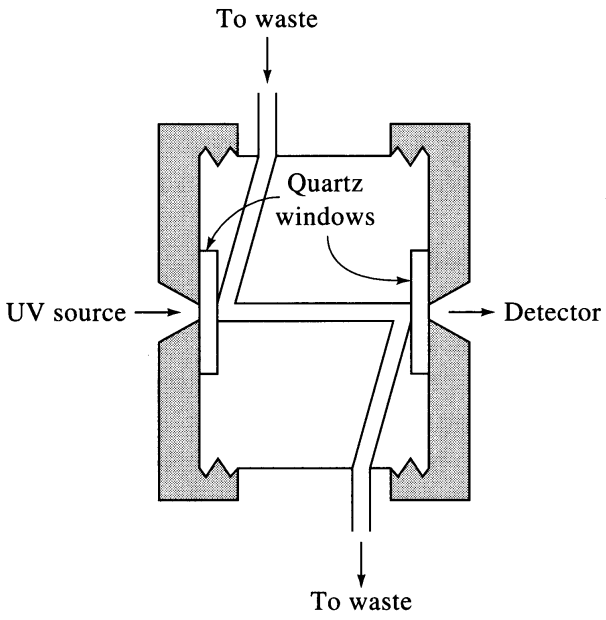
(i) Small tube diameters (<0.1-1 mm id)

(ii) Reactor coil (<50 cm long) tightly wound to increase mixing

Detection:

(i) Photometer (single λ)

(ii) UV-vis spectrophotometer (multichannel detection)



(iii) Electrochemical (potentiometry)

(iv) Atomic spectroscopy (AES or AAS)

Instrumentation can be quite complex:

Example: phase mixing

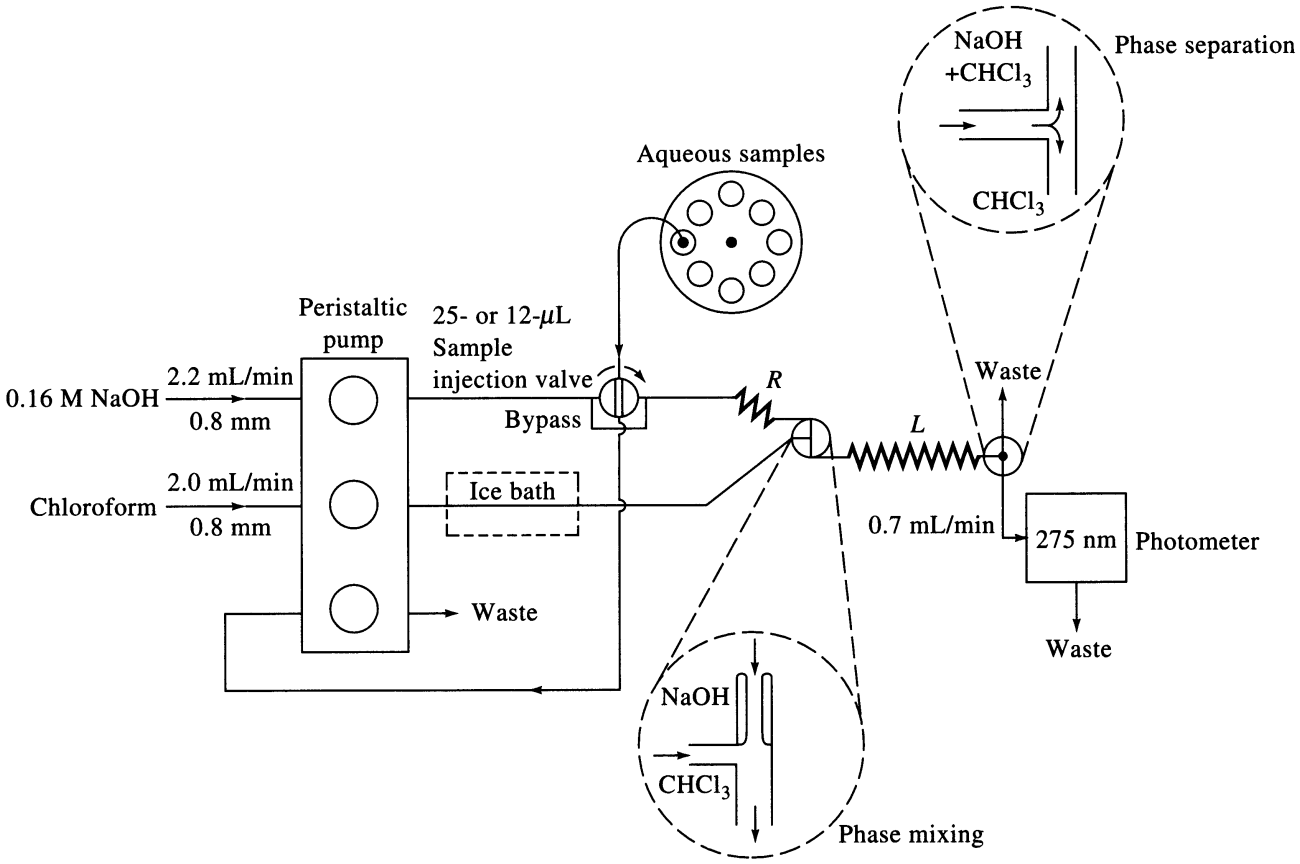


Fig 33-8

Quantifying Diffusional Processes in FIA

Dispersion:

concentration in injection volume

$$D = \frac{c_0}{c}$$

peak concentration at detector

Calibration needed to find D

D affected by

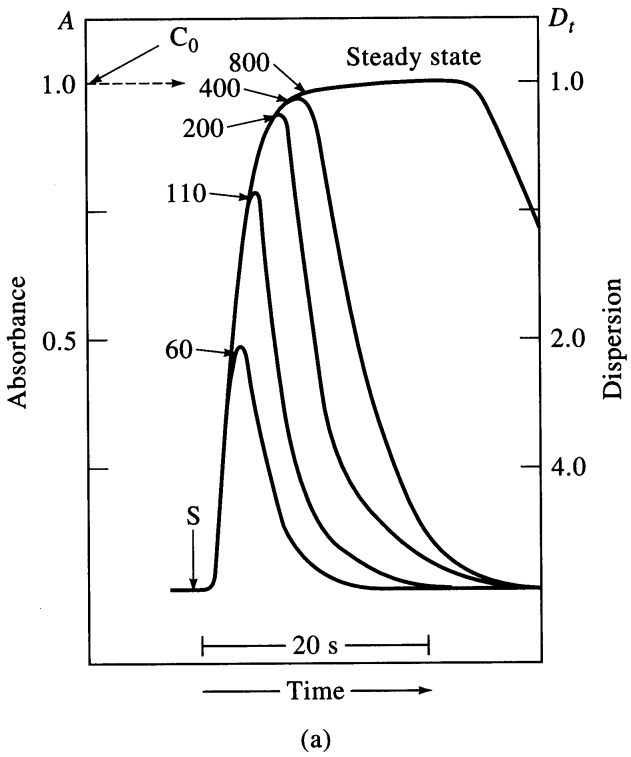
(i) sample volume

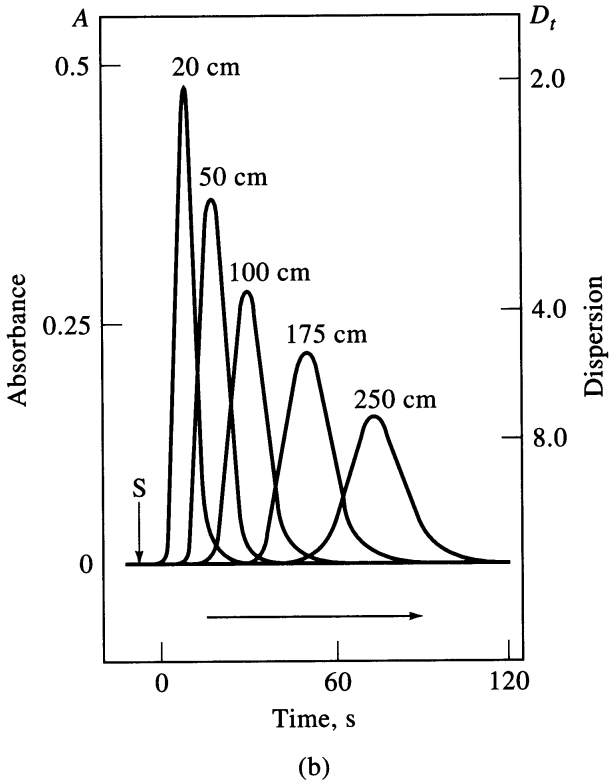
(ii) tube length

(iii) flow rate

(iv) tube id

Fig 33-6





Large injection volume: D = 1.0 (no analyte dilution by carrier)

Low volume: D > 1.0 (analyte dilution)

Short tubes: less time for diffusion = low dispersion

Long tubes: long time for diffusion = high dispersion

Limited Dispersion (D=1-3) FIA:

- Short reactors tubes
- Small tube id's
- Medium flow rates - fast (>300 samples hr^{-1})
- Large sample volumes

Medium Dispersion (3-10) FIA:

- Suited to slower reactions rates
- Longer reactors
- Lower flow rates
- Small sample volumes - sensitive

Large Dispersion (D>10) FIA:

- Best for reactions needing time to equilibrate
- Long reactors
- Low flow rates - slow
- Small sample volumes but high dispersion - less sensitive
- Not used much