Supplement #3: Gas Chromatography

Michael Tswett

Traditional Packed Column- Liquid Chromatography (ala' Tswett)

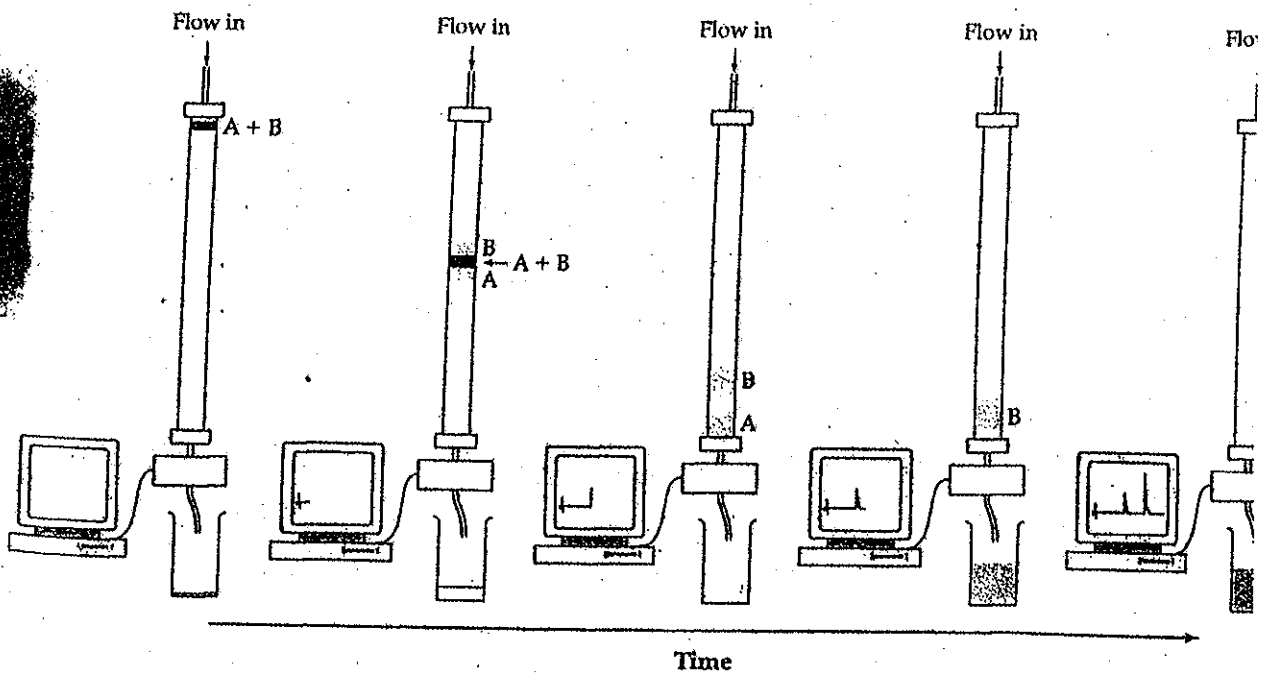
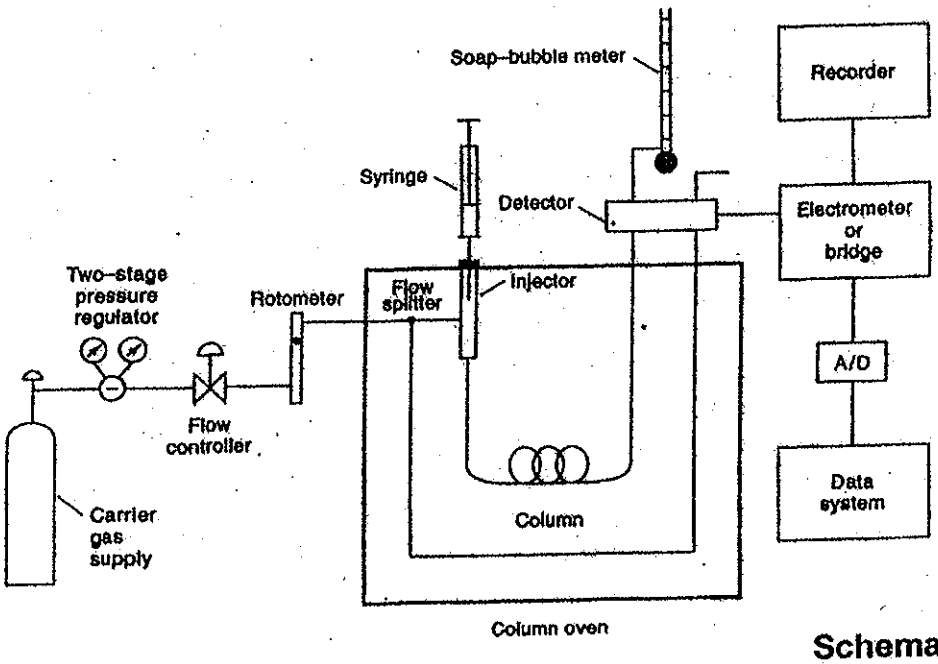


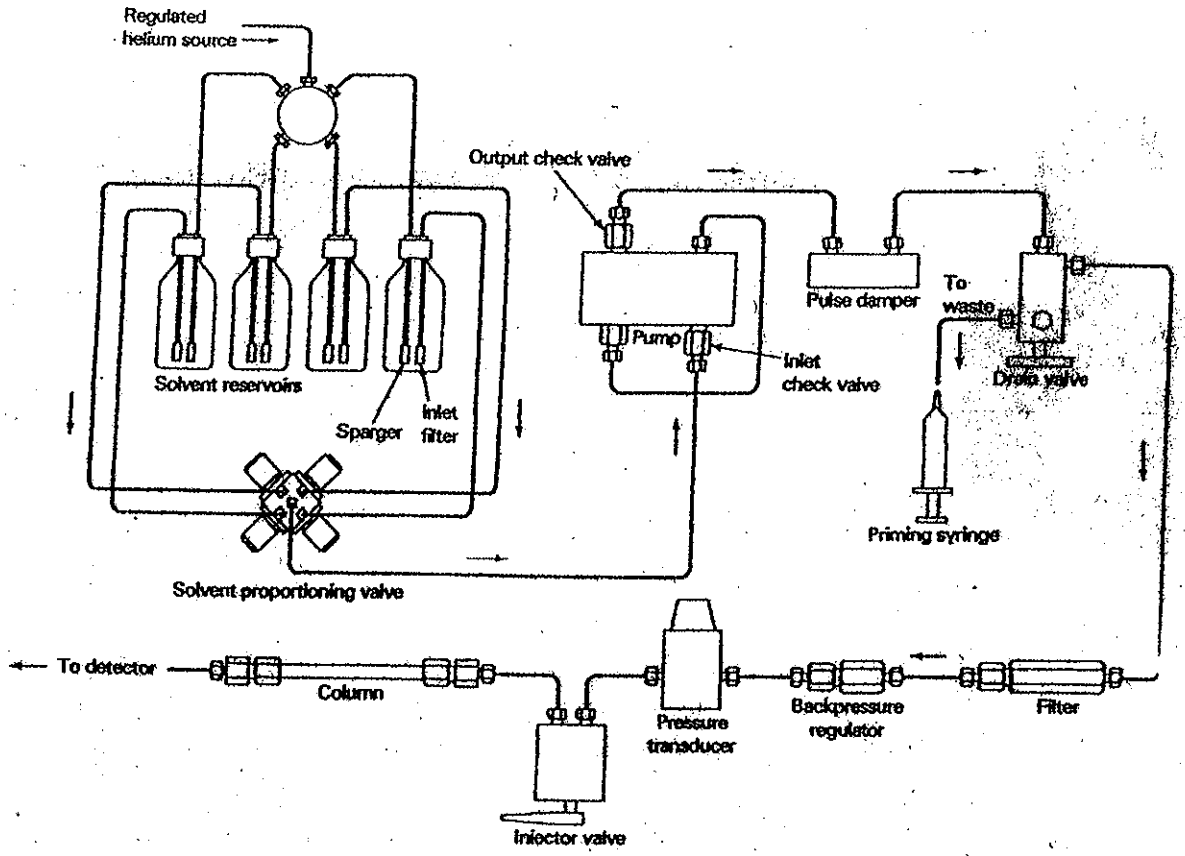
FIGURE 13.1 ▲
Illustration of column-liquid chromatography.

The system is shown at different times (not at regular intervals) after the sample enters the column. The column is packed with a solid adsorbent. The sample, composed of mixed components A and B, is carried through the column by the flowing liquid. As they pass the detector, their presence is recorded on the data system. Note that the concentrations of A and B as they exit the column are always lower than when injected.



Schematic of Gas Chromatograph (GC)

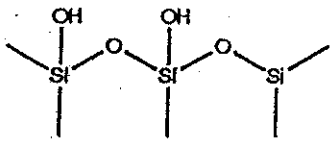
Schematic of High Performance (Pressure) Liquid Chromatography (HPLC)



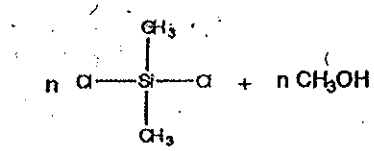
Common Terms in Chromatography

Mobile Phase	-medium carrying sample across
Stationary Phase	-site of separation-usually packed or coated on column
Support	- the inert surface on which the stationary phase is bonded
HETP	-Height Equivalent of Theoretical Plate=the effective distance between distinct separation jumps
Normal phase	-LC short hand for case of non-polar mobile phase and polar stationary phase
Reversed phase	-LC shorthand for case of polar mobile phase and non-polar stationary phase (set up in current Alfred HPLC)
Resolution	-A numeric estimate of the degree of peak separation.
Capillary column	-Also called open tubular column, a thin glass capillary with the stationary phase coated on the inside walls
Packed column	-traditional ~ ¼ inch metal columns tightly packed with stationary phase on finely divided silaceous support.

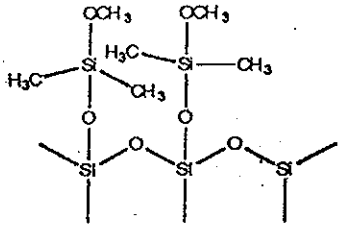
Silanizing the support



hydrolyzed support surface



dimethylchlorosilane (DMCS)



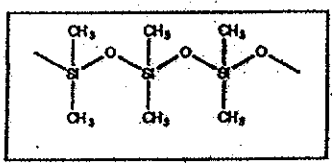
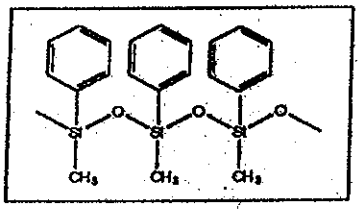
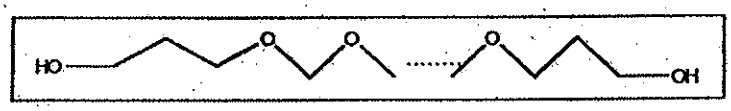
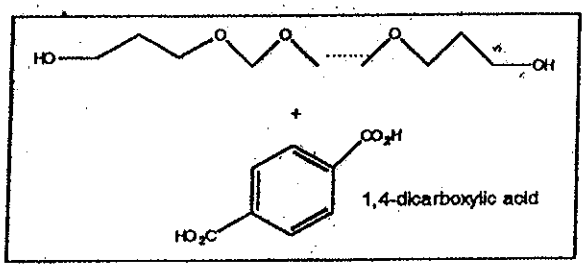
+n HCl

hydrolyzed support surface

Tends to adsorb polar organic components like ROH and water, causing degradation of separation

Silanized support won't bleed or causing tailing

Examples of Liquid Phases Used to Coat the Support

Chemical Name	commercial name	Chemical Structure	Uses
Polymethyl silicone	OV-1,SE-30		general purpose Non-polar samples (aromatics, aliphatics, PCBs, drugs, steroids
Poly(phenymethyl) siloxane (5o:50 methyl:pheny)	OV-17		aromatic compounds glycols, steroids
Polyethylene glycol	Carbowax 20M		acids, ethers, natural oils
Polyethylene glycol-terephthalic acid	OV-351		alcohols, fatty acids

TCD (thermocouple detector)

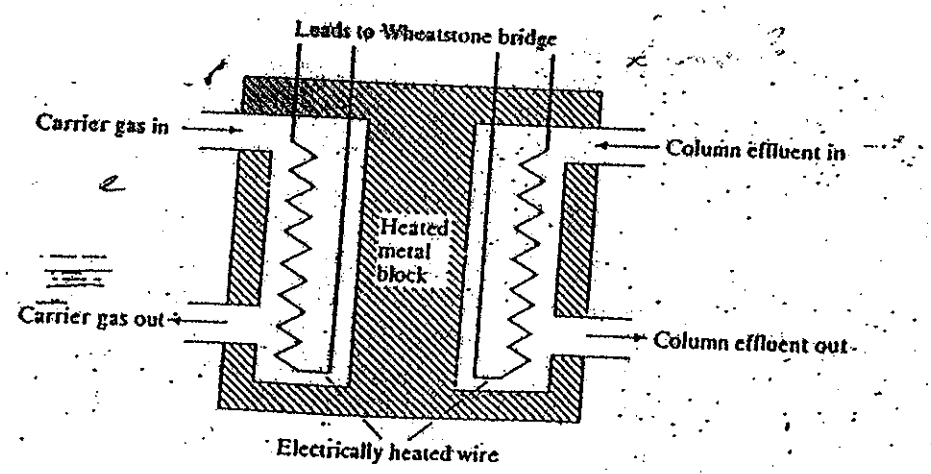
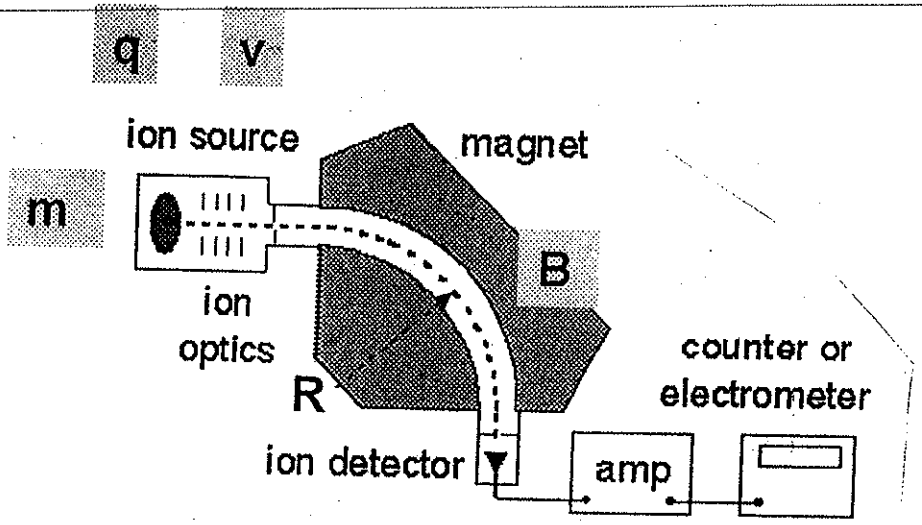


Figure 26-7 Diagram of a two-filament thermal conductivity detector.

Voltage-based detector

Mass spectrum detector (GC-MS)



SIDE A (carbowax)

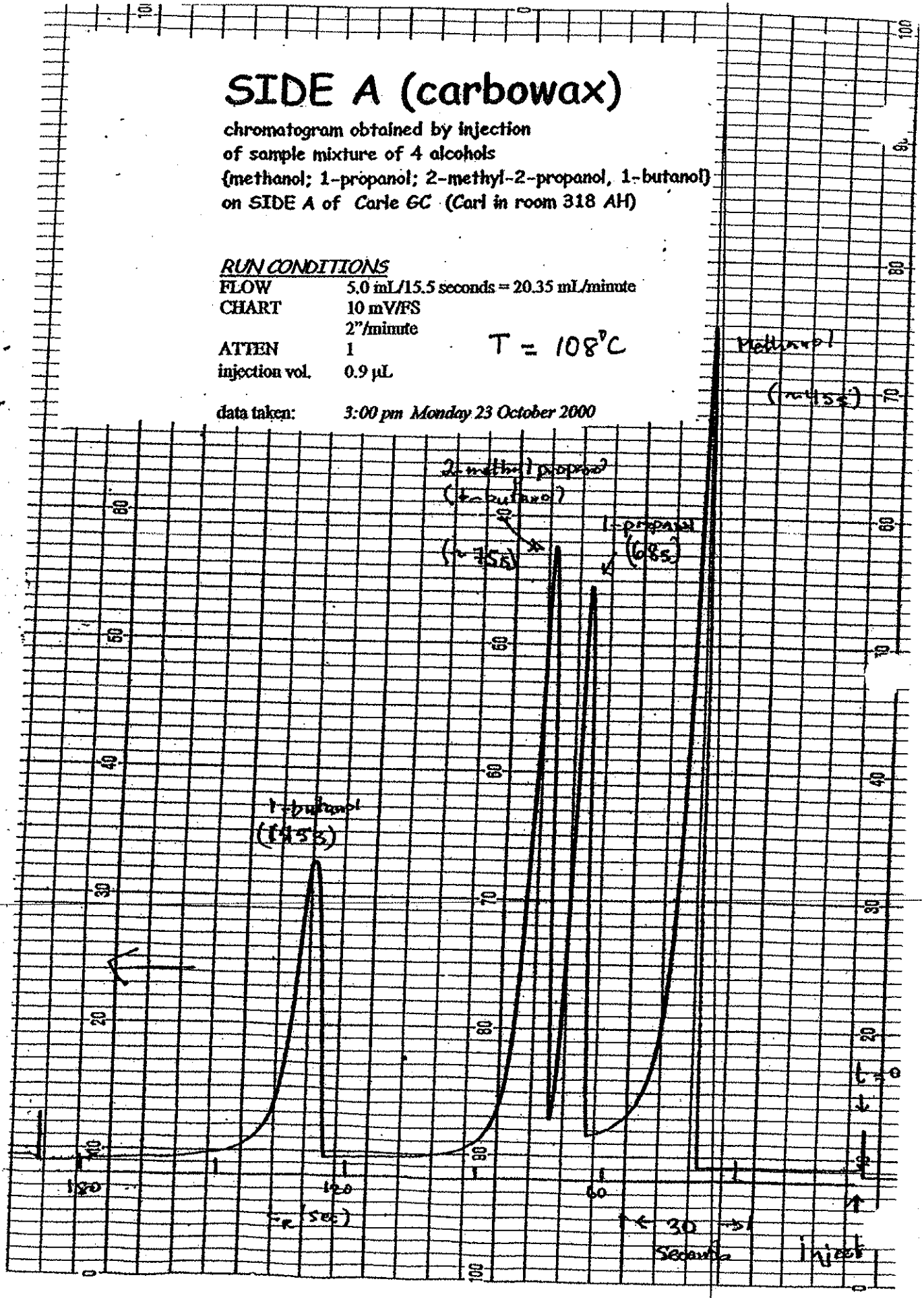
chromatogram obtained by injection
of sample mixture of 4 alcohols
(methanol; 1-propanol; 2-methyl-2-propanol, 1-butanol)
on SIDE A of Carle GC (Carl in room 318 AH)

RUN CONDITIONS

FLOW	5.0 mL/15.5 seconds = 20.35 mL/minute
CHART	10 mV/FS
	2"/minute
ATTEN	1
injection vol.	0.9 µL

T = 108°C

data taken: *3:00 pm Monday 23 October 2000*



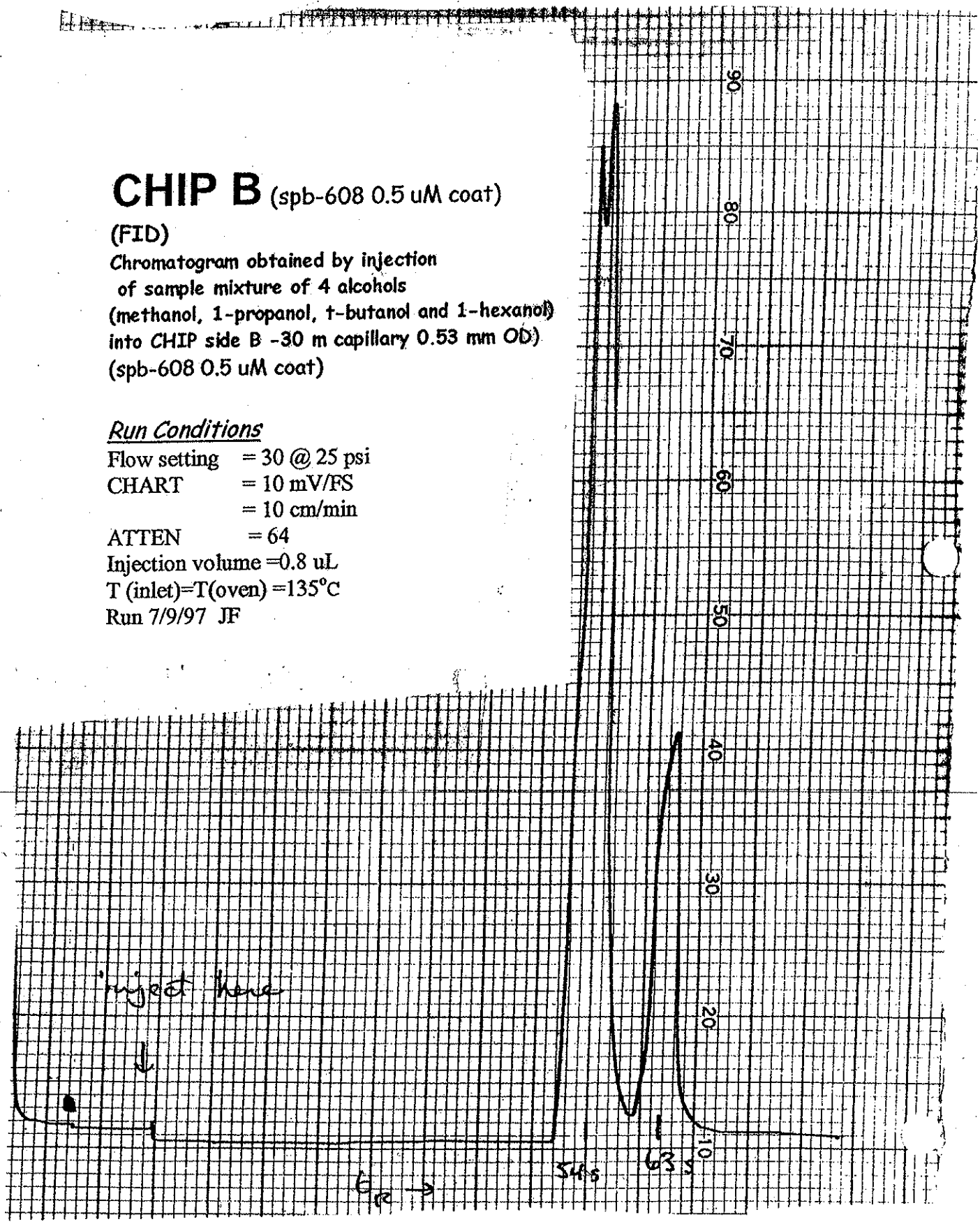
CHIP B (spb-608 0.5 uM coat)

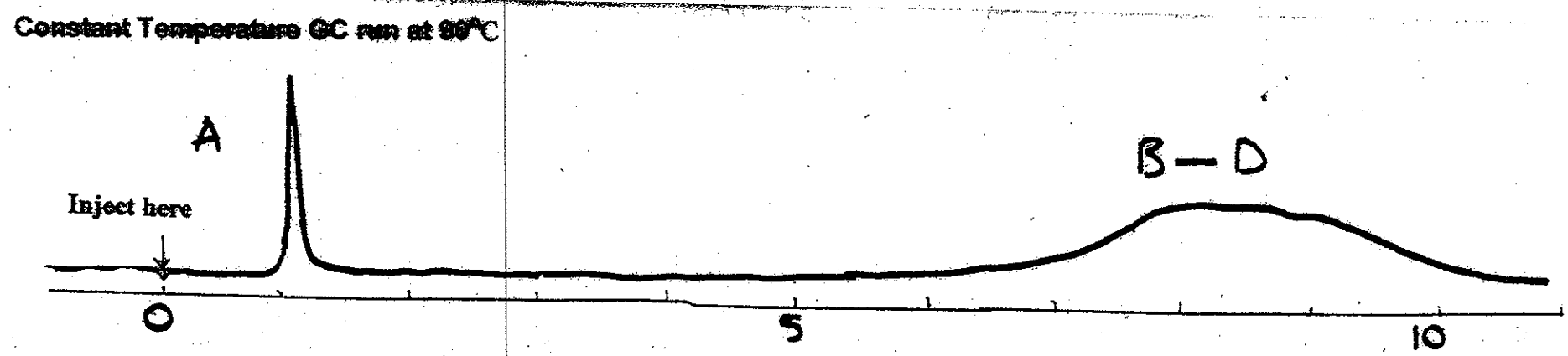
(FID)

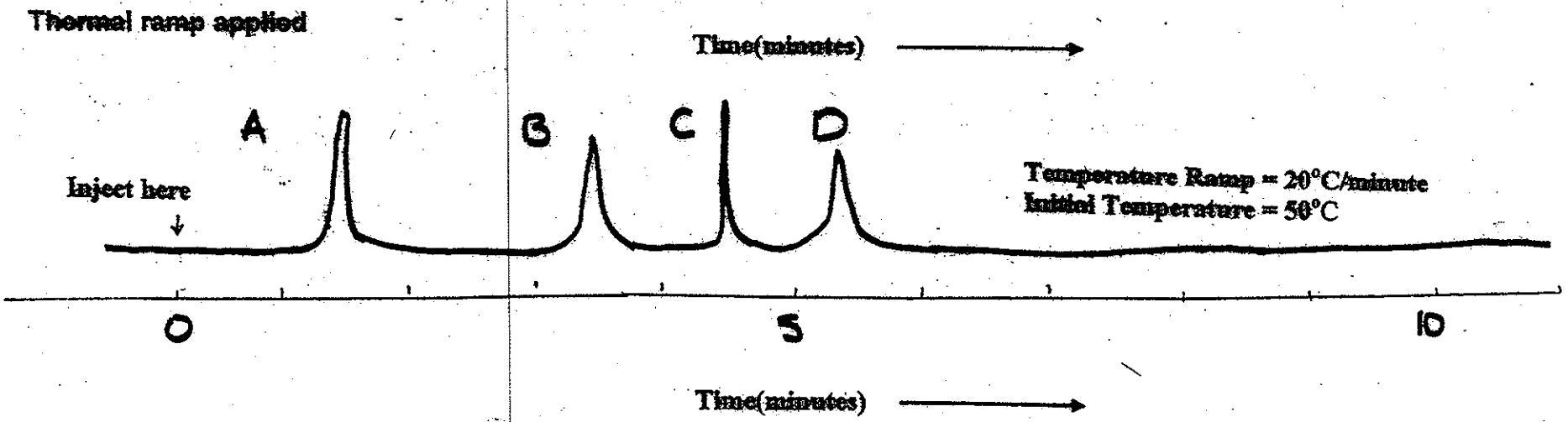
Chromatogram obtained by injection of sample mixture of 4 alcohols (methanol, 1-propanol, t-butanol and 1-hexanol) into CHIP side B -30 m capillary 0.53 mm OD) (spb-608 0.5 uM coat)

Run Conditions

Flow setting = 30 @ 25 psi
CHART = 10 mV/FS
= 10 cm/min
ATTEN = 64
Injection volume =0.8 uL
T (inlet)=T(oven) =135°C
Run 7/9/97 JF







Composition Of Injected Mixture Vs Bp And GC Behavior Under Temperature Ramp

Component	boiling point, °C	t_R(with ramp)	T(oven) at t_R
A	80	1.5 min	80
B	125	3.3	116
C	140	4.4	138
D	160	5.3	186

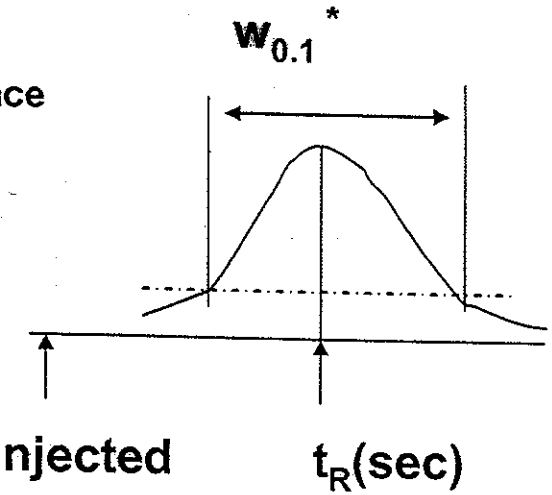
Computing number of plates N and length of plates H to a Column of Length L

Synge/Martin theory connection to reality of GC performance

$$H = L(cm)/N$$

For perfectly symmetric peaks (Z=1)**

$$N = 18.5(t_R/w_{0.1})^2 = \#\ plates$$



Symmetric peak

For asymmetric peaks (Z ≠1)

$$N = \frac{41.7(t_R/w_{0.1})^2}{[Z + 1.25]} = \#\ plates$$

Asymmetric peak

* $w_{0.1}$ is width at 1/10 the max height of peak

*$Z = w_R/w_L$ (ratio of right to left side of $w_{0.1}$ defined by peak) see also supplement 14

Typical calculation for **N** and H

L=Column length = 6 feet ~ 183 cm

t_r= 3.0 minutes =180 sec

$w_{0.1}$ = 0.15 minutes= 9 seconds

Z=1 (symmetric, `pretty' peak)

$N=18.5(t_R/w_{0.1})^2$=# plates

=18.5(180/6)2 = 16650

H= L(cm)/N

H= 183/16650 ~ 0.011 cm