

37.1 THIN LAYER CHROMATOGRAPHY

Learning outcomes

Candidates should be able to:

1. describe and understand the terms
 - (a) stationary phase, for example aluminium oxide (on a solid support)
 - (b) mobile phase; a polar or non-polar solvent
 - (c) R_f value
 - (d) solvent front and baseline
2. interpret R_f values
3. explain the differences in R_f values in terms of interaction with the stationary phase and of relative solubility in the mobile phase

Introduction

Thin Layer Chromatography (TLC) is a technique used to analyse small samples via separation

There are 2 phases involved in TLC:

1. Stationary phase

This phase is commonly a thin metal sheet/glass slide coated in alumina (Al_2O_3) or silica (SiO_2).

The solute molecules **adsorb** onto the surface.

Depending on the strength of interactions with the stationary phase, the separated components will travel particular distances through the plate.

The more they interact with the stationary phase, the more they will 'stick' to it.

2. Mobile phase

Flows over the stationary phase.

It is a polar or nonpolar liquid (solvent) or gas that carries components of the compound being investigated

- Polar solvents - water or alcohol
- Non-polar solvents - alkanes

If the sample components are coloured, they are easily identifiable

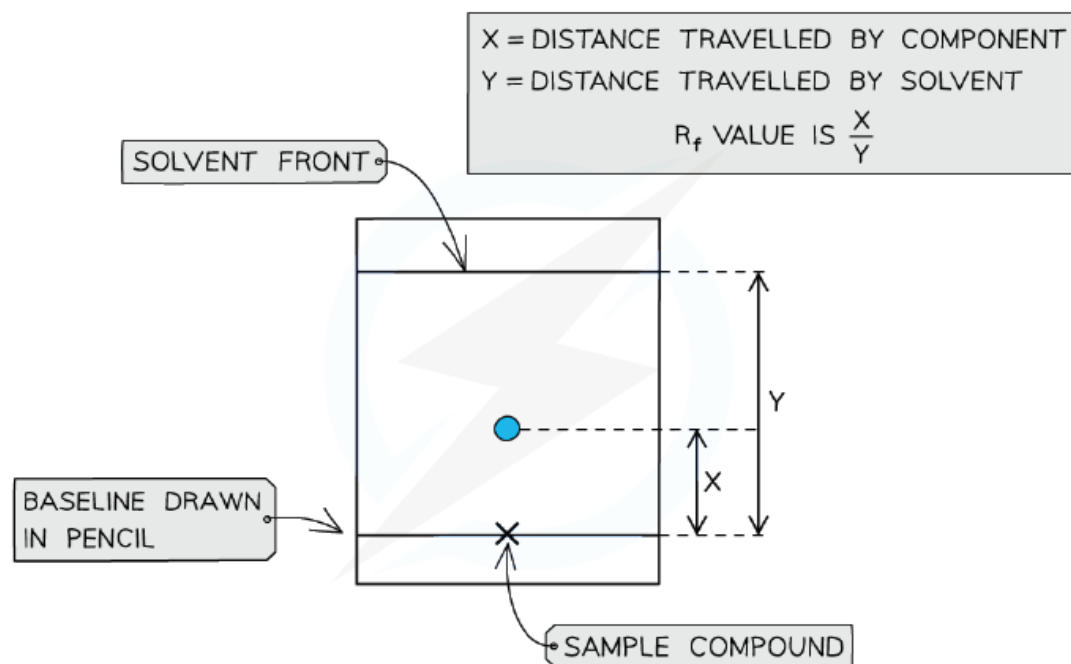
We can examine the plate under UV light using ninhydrin to identify uncoloured components

R_f values

A TLC plate can be used to calculate R_f values for compounds:

$R_f = \text{distance travelled by component} / \text{distance travelled by solvent}$

These values can be used alongside other analytical data to deduce the composition of mixtures



The less polar components travel further up the TLC plate

- Their R_f values are higher than those closer to the baseline
- They are more soluble in the mobile phase and get carried forward with the solvent

More polar components do not travel far up the plate, they are more attracted to the polar stationary phase

The extent to which the components within the investigated sample are separated depends on:

- The solubility of each component in the mobile phase
- The interaction between each component and the stationary phase

Knowing the R_f values of compounds being analysed, helps to compare the polarity of various molecules.

Thin Layer Chromatography (TLC)

1. Thin-layer and gas / liquid chromatography can be used to separate mixtures into their individual components. Define the following terms used in chromatography.

R_f value

.....

retention time

.....[2]

2. A mixture of two substances A and B is analysed by thin-layer chromatography. The R_f value of substance A is larger than that of substance B. Suggest why substance A has a larger R_f value.

.....

..... [1]

3. A student uses thin-layer chromatography (TLC) to analyse a mixture containing different metal cations. The student repeats the experiment using different solvents. Fig. 6.1 shows the chromatogram obtained by the student using water as a solvent.

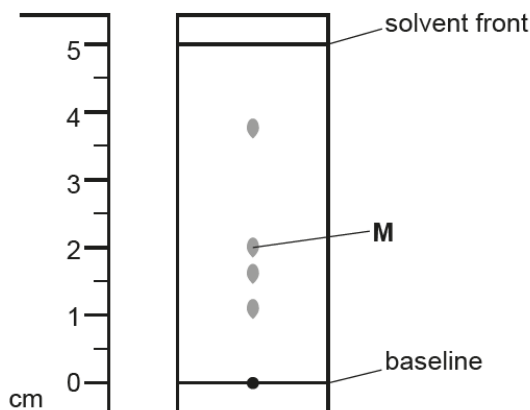


Fig. 6.1

(a) (i) Suggest a compound that could be used as the stationary phase in this experiment.

..... [1]

(ii) Table 6.1 shows the R_f values for different metal cations when separated by TLC using water as a solvent.
Table 6.1

Cation	R_f value (water)
Cd^{2+}	(aq) 0.40
Co^{2+}	(aq) 0.77
Cu^{2+}	(aq) 0.32
Fe^{3+}	(aq) 0.12
Hg^{2+}	(aq) 0.23
Ni^{2+}	(aq) 0.75

Suggest the identity of the cation that causes the spot at **M** in Fig. 6.1.
Explain your answer.

.....
..... [1]

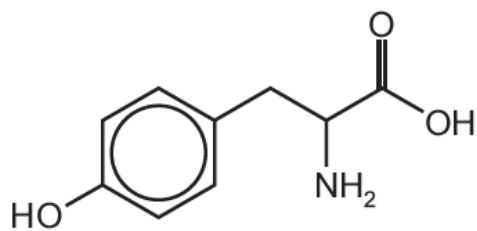
(b) The student repeats the experiment using butan-1-ol as a solvent.
The metal cations do **not** travel as far up the TLC plate in this experiment.
Suggest why the metal cations do not move as far up the TLC plate with butan-1-ol as a solvent.

.....
..... [1]

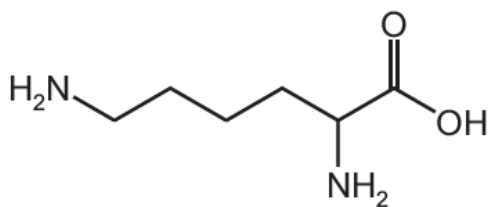
(c) The student sprays the TLC plate in Fig. 6.1 with KSCN(aq) .
The colour of some of the spots changes, as some of the metal cations undergo a ligand exchange reaction.
Identify the ligands involved in the ligand exchange reaction.

..... exchanges with [1]

4. Tyrosine and lysine, shown in Fig. 9.1, are naturally occurring amino acids found in proteins.



tyrosine



lysine

Fig. 9.1

A mixture of tyrosine and lysine can be separated by thin-layer chromatography. Under certain conditions the R_f value of lysine is 0.14 and the R_f value of tyrosine is 0.45.

- (i) Explain what is meant by R_f value.

[1]

- (ii)** Suggest an explanation for the difference in R_f values.

[1]

5. The purity of lidocaine can be checked using thin-layer chromatography.

Ethyl ethanoate is used as the solvent.

The R_f values of **X** and lidocaine are given in Table 6.1.

Table 6.1

Compound	<i>R</i> _f
X	0.49
lidocaine	0.71

- (i)** Identify the substances used as the mobile and stationary phases in this thin-layer chromatography experiment.

mobile phase

stationary phase [1]

- (ii) Describe how an R_f value can be calculated.

[1]

- (iii) Suggest why the R_f value for **X** is less than that for lidocaine.

[1]

*****This is not a complete note. It is to guide you. It is recommended to study the prescribed textbooks along with this material. *****