



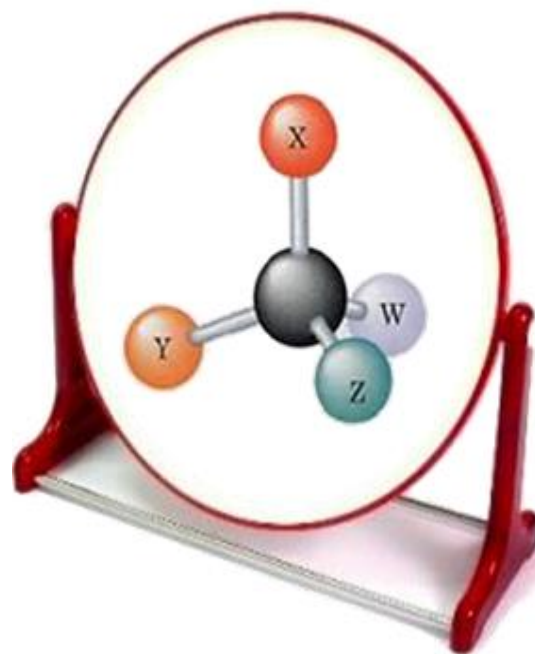
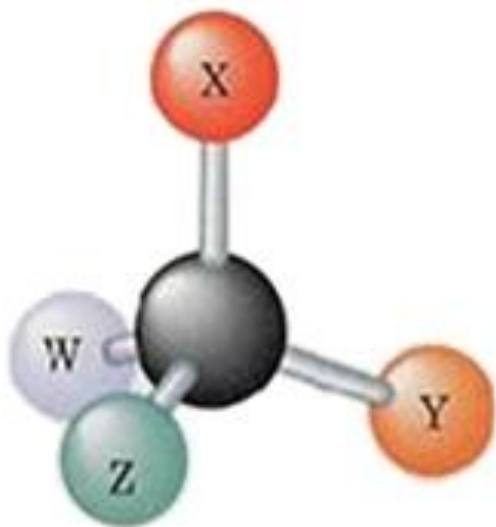
外消旋化合物的分離

Resolution of Racemic Phenylsuccinic Acid
Using (-)-Proline as a Resolving Agent

<http://orglab.thu.edu.tw>



1. Enantiomers



2. (+,-) System

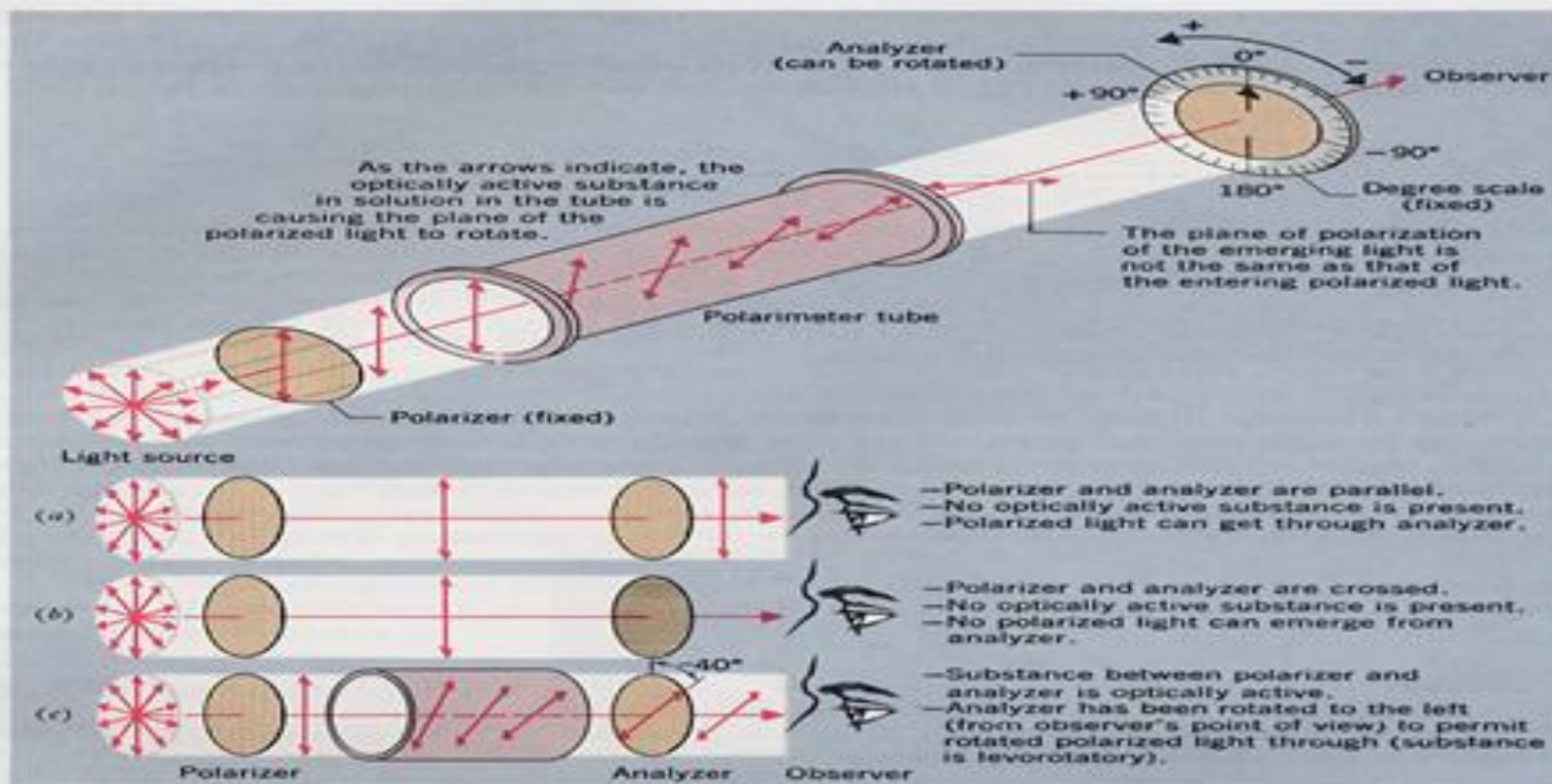
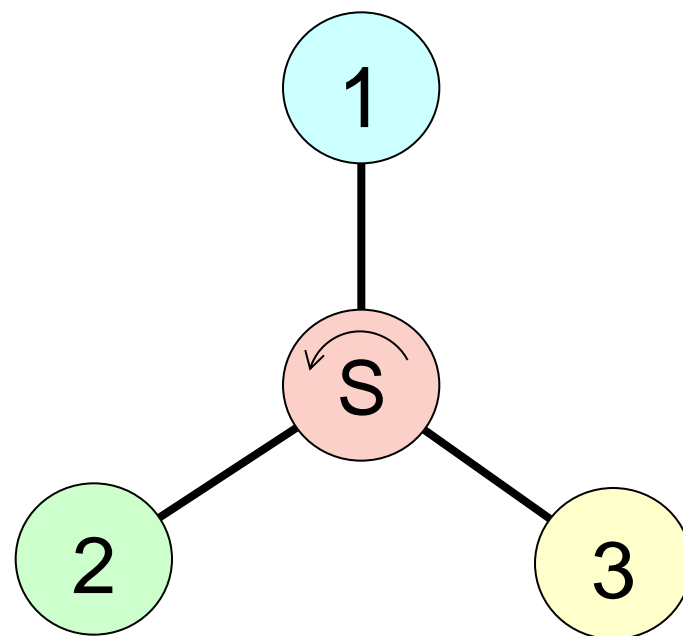
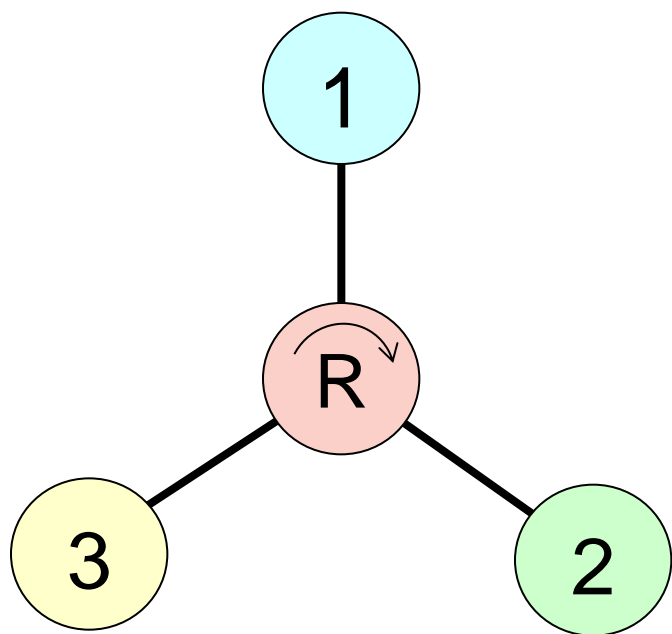


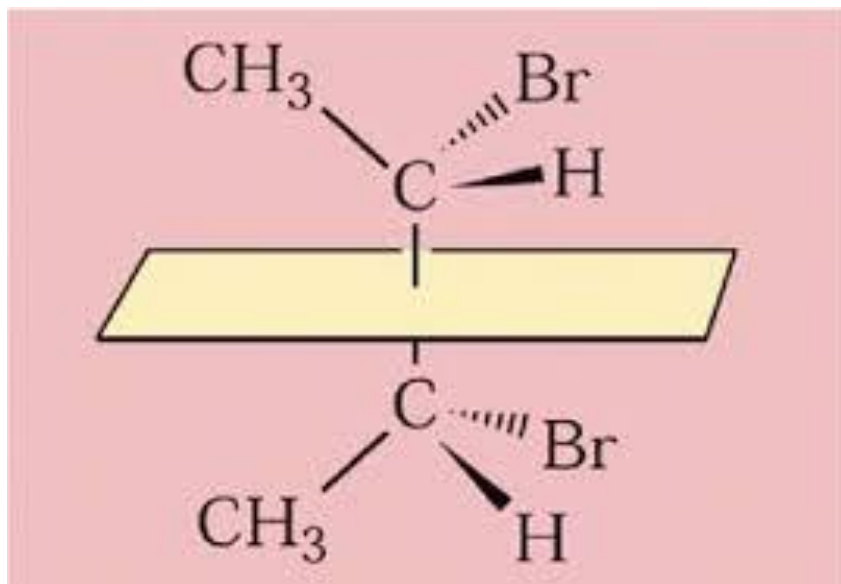
FIGURE 5.11 The principal working parts of a polarimeter and the measurement of optical rotation. (From John R. Holum, *Organic Chemistry: A Brief Course*, Wiley, New York, 1975, p. 316.)

3. R-S System



4. Racemic \rightarrow 50% R + 50% S

5. Meso compounds



The plane of symmetry of meso-2,3-dibromobutane.

6. Specific rotation: (旋光度)

$$[\alpha]_{\lambda}^t = \frac{\alpha}{l \times c}$$

$[\alpha]$ = specific rotation

t = temperature in degrees Celsius

λ = wavelength of incident light

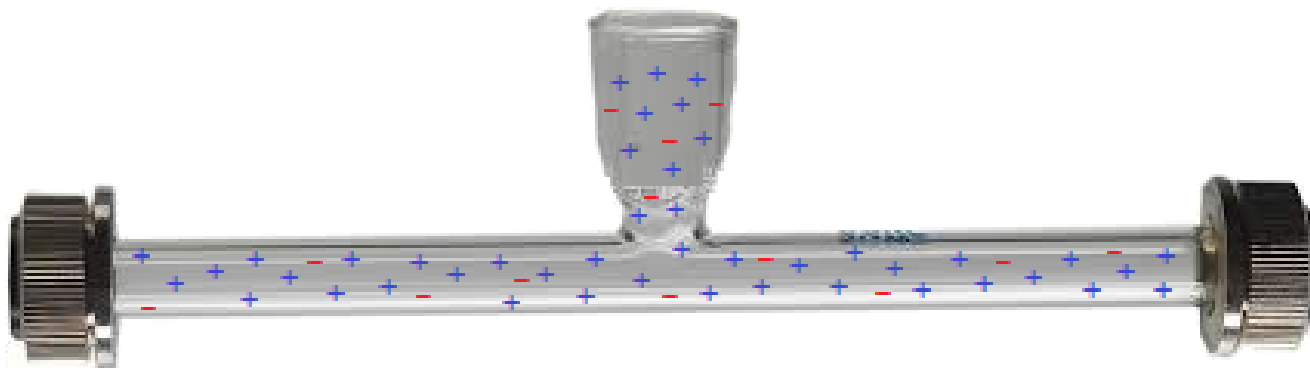
α = observed optical rotation in degree

l = length of sample container in decimeters
(1dm=10cm)

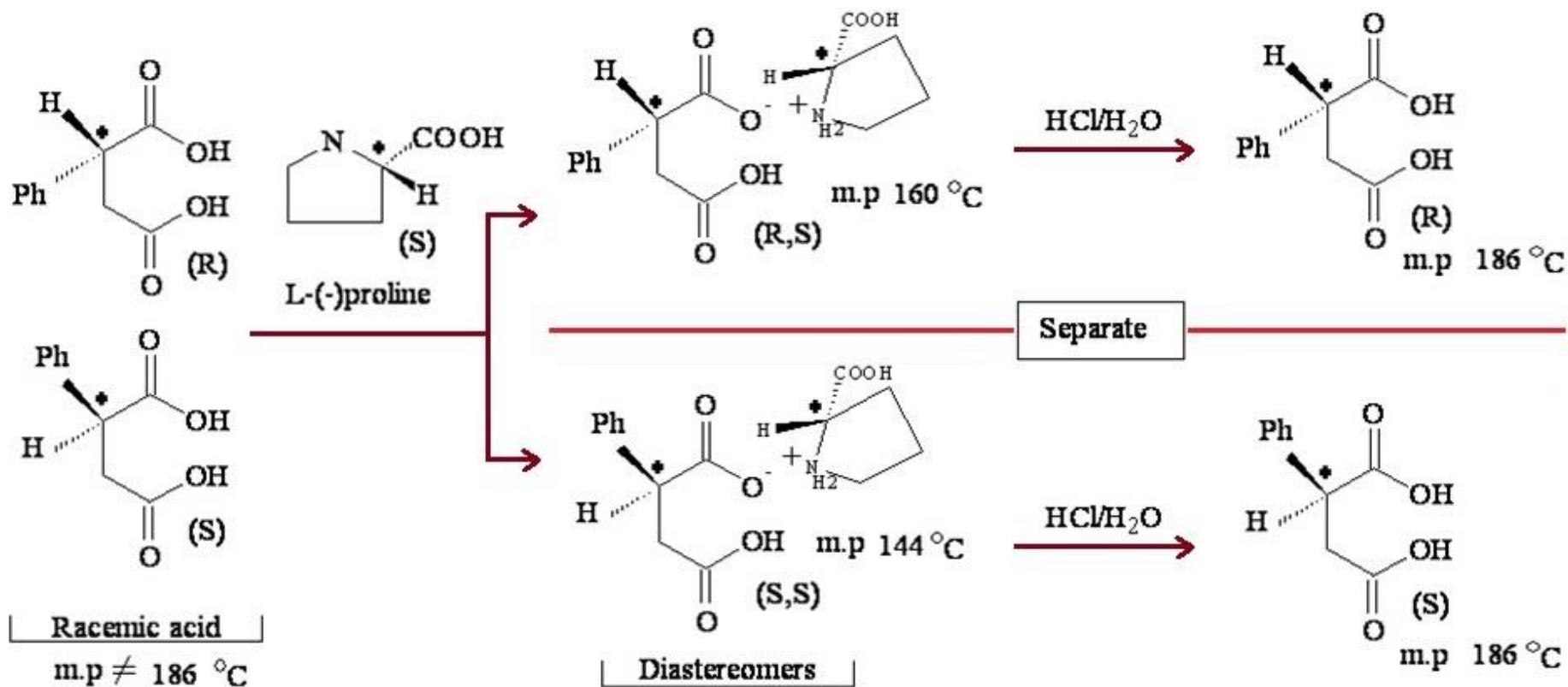
c = concentration (grams per milliliter of solution)

7. 分離純度:

* (+)-2-Phenyl Succinic acid 分離純度: 設為x
則 $180^\circ x + (-180^\circ)(1-x) = [\alpha]$



* (-)-2-Phenyl Succinic acid 分離純度: 設為y
則 $180^\circ(1-y) + (-180^\circ) y = [\alpha]$



**0.65g (+,-)-phenylsuccinic acid + 17ml iso-Propanol
in 50ml r.b.d flask**

↓
swirl to dissolve

↓
add L-(-)-proline(0.38g) in one portion

↓
swirl the mixture for 2-3 min

↓
reflux for 20min

↓
cool to approximate 30°C

↓
**filter and wash the solid twice with 5 ml Acetone
(need collect the filtrate)**



2. (S)-(+)-Phenylsuccinic acid proline salt

put it in oven to dry completely**

↓
add into a pre-cooled flask contain 6M HCl (5mL)

↓
swirl the mixture for 5 min

↓
filter and wash the solid twice with 5mL cold water
(S)-(+)-Phenylsuccinic acid as white solid

↓
dry
↓
weight

3. Isolation of (R)-(-)-Phenylsuccinic acid filtrate

- ↓
evaporate
- ↓
add 6M HCl (5mL)
- ↓
swirl for 10 min
- ↓
filter and wash the solid twice with 5mL cold water
- ↓
dry
- ↓
weight

4. measure specific rotation:
(pure compound $[\alpha]=180^\circ$)

dissolve 0.1g sample in 10 mL acetone

↓
put the solution into sample container

↓
measure and record α value

↓
calculate $[\alpha]$



1. 需在儀器使用登記表上記錄旋光度並簽名。
2. 記錄並告知分離產物各別淨重。
3. 實驗問題：1, 2



Thank you !

<http://orglab.thu.edu.tw>

