

THE BASIC PRINCIPLE OF MASS SPECTROSCOPY & ITS INSTRUMENTATION

Muthyala Bala Krishna
Asst. Professor
Pharmaceutical Analysis
VIPW

Contents



Introduction

Basic principle

Advantages & Disadvantages

Instrumentation

Applications

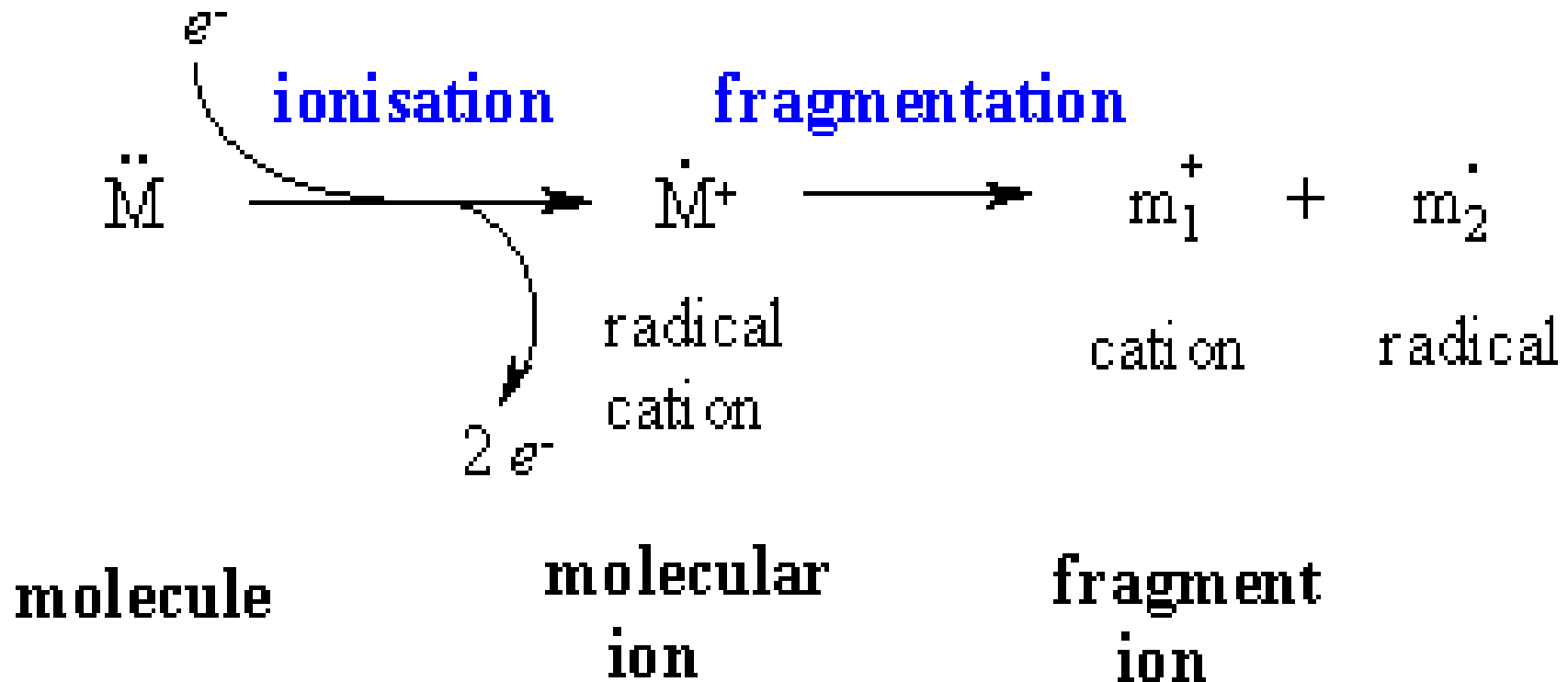
INTRODUCTION

- Mass spectroscopy deals with the study of the charged molecules & fragment ions produced from a sample exposed to ionizing conditions, & also of the relative intensity spectrum which results from the correlation of the ions with their mass to charge ratio.
- MS concept is first introduced by scientists Sir.J.J. Thomson and Wiens.



Principle

When gaseous sample under 10^{-7} to 10^{-5} mm Hg pressure bombarded with electron beam(70eV), loss of electron from π orbital takes place resulting in the formation of ions and ion fragments.



Types of ions

Molecular ions

Fragment ions

Rearrangement ions

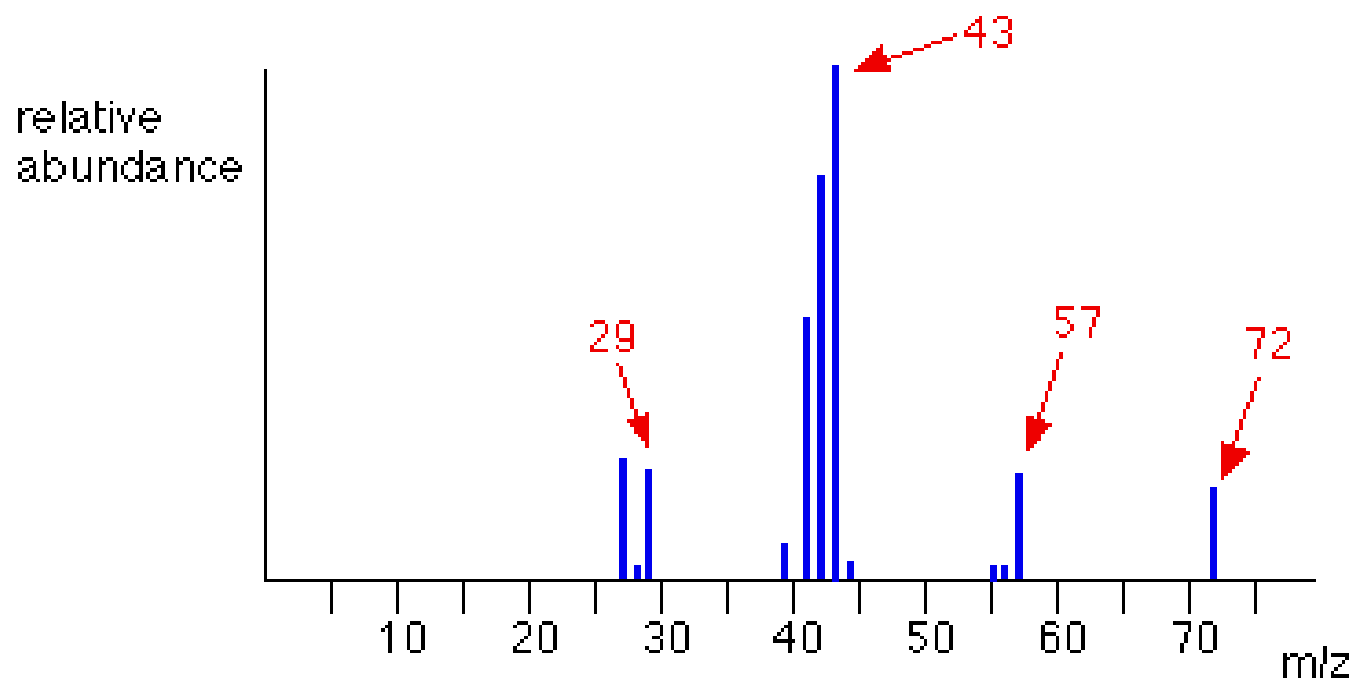
Multiple charged ions

Meta stable ions

Negative ions

Isotopic ions

simplified mass spectrum of pentane - $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$



Advantages & disadvantages

Molecular weight & formula determination

Qualitative & Quantitative analysis

Less amount of sample

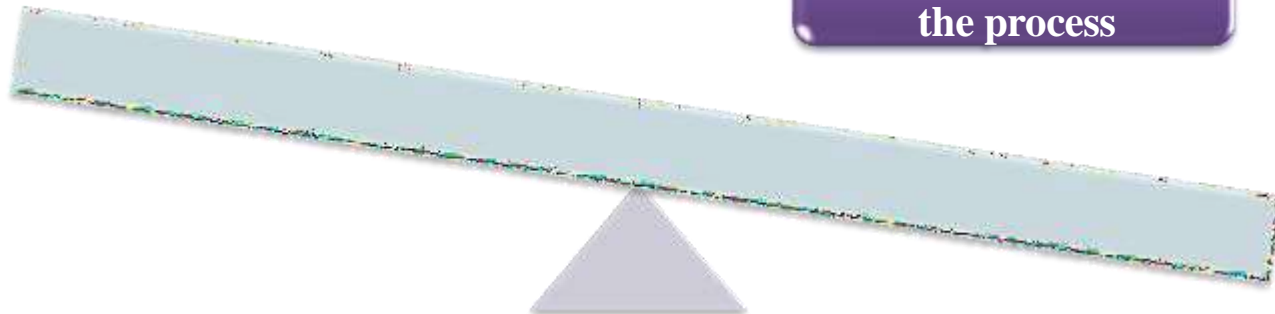
Less than 1min. for analysis

Sample destruction

Sample should be in gaseous form

Complex & high cost

Should maintain vacuum throughout the process

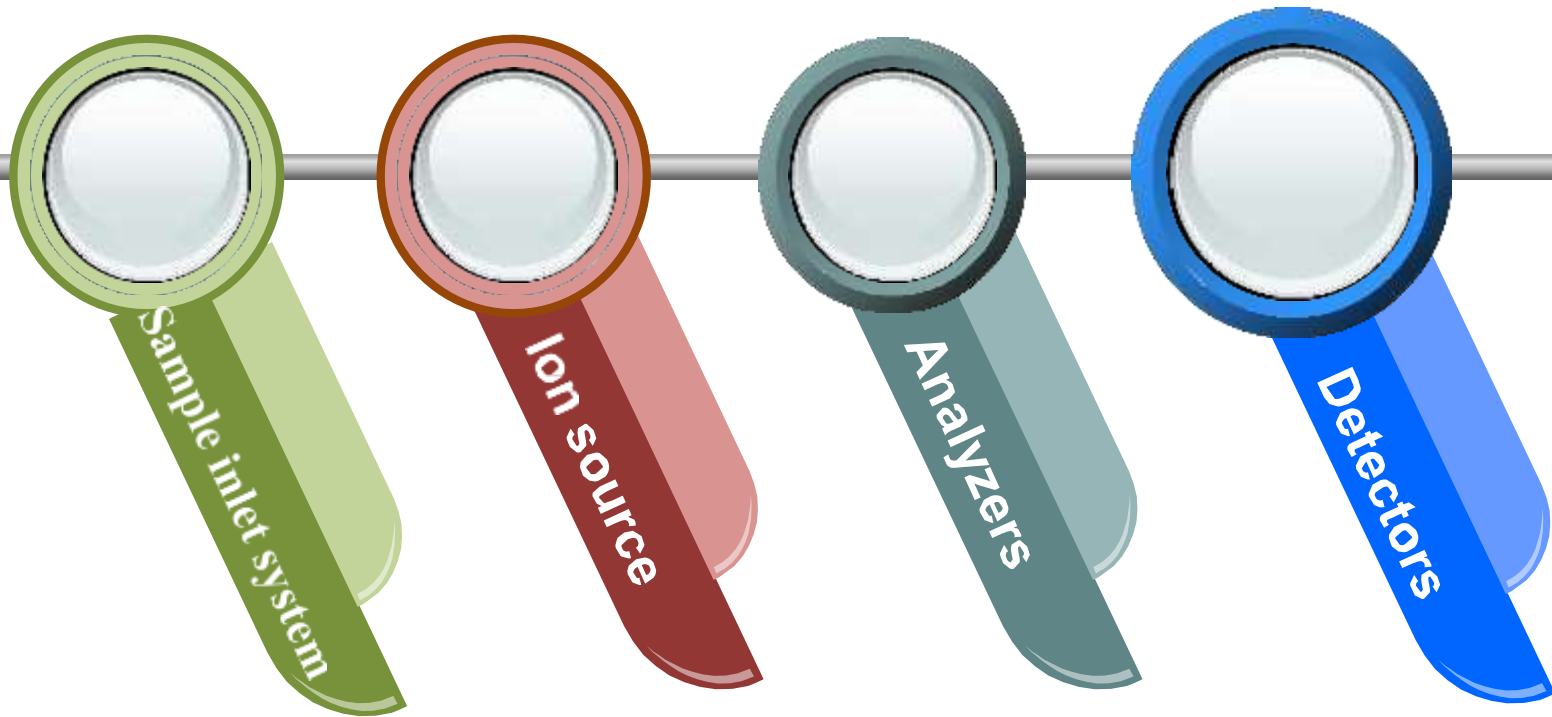


INSTRUMENTATION

Mass Spectrometer



BASIC COMPONENTS



SAMPLE INLET SYSTEM

solid

Directly into the chamber with low vapour pressure by means of probe.
e.g. steroids, carbohydrates

Liquid

Vaporized with the help of heat inlet system.
e.g. sugars , amino acids

Gas

Directly inleted & vaporized

Ionization techniques



**Electron
impact
ionization**

**Chemical
ionization**

**Field
ionization**

**Laser
desorption
technique**

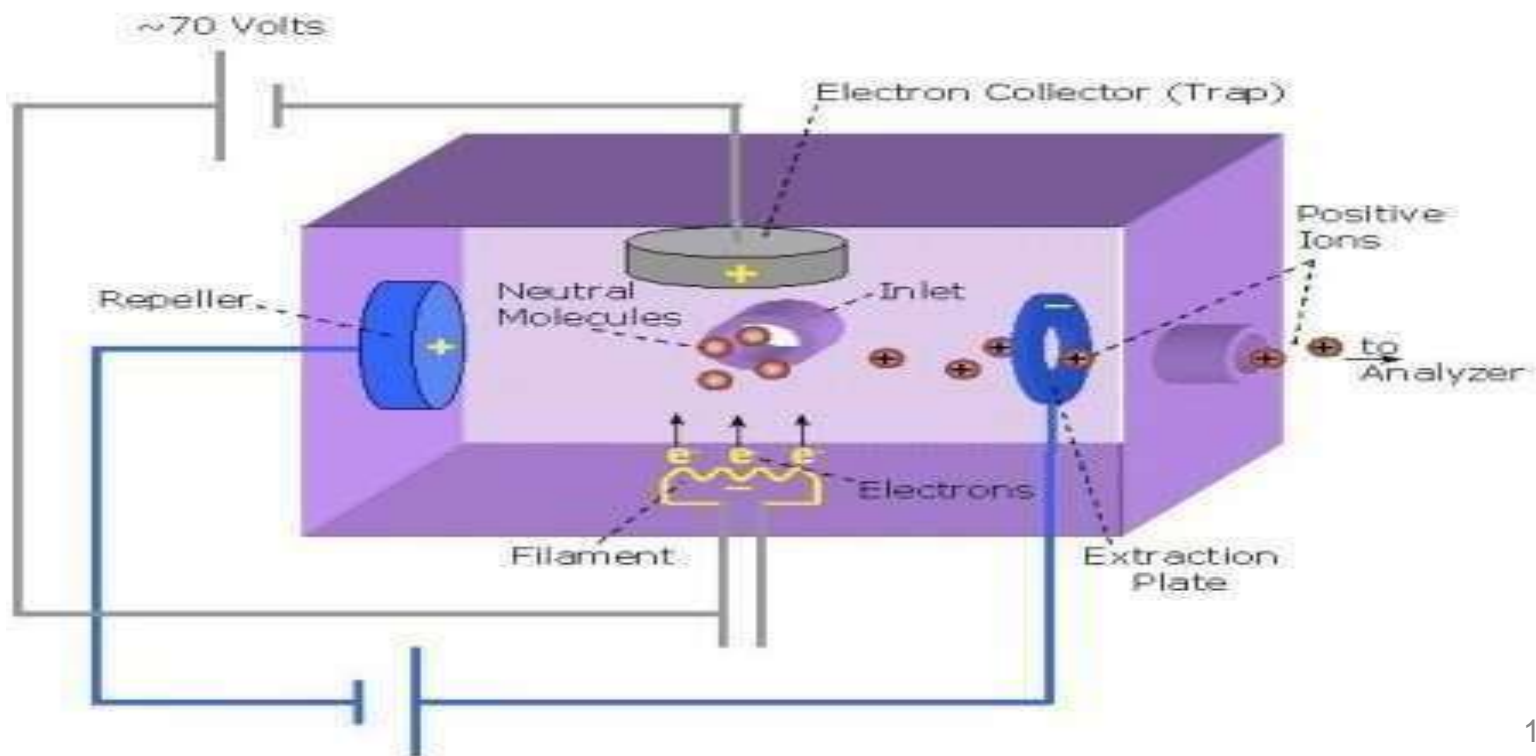
**Fast acting
bombardment**

Electron impact Ionisation:

Electron beam(70ev) produced from electrically heated tungsten or rhenium filament is applied perpendicular to the sample and results in fragmentation.

*sample destruction.

Electron Impact Ionization Source



Chemical ionization:

Reactive gases bombards with
electron beam



Reactive species are formed which
colloid with sample molecules



+vely charged ions are produced

Fast atom bombardment:

Fast atoms
of Xe/Ar
collides
with neutral
gas atoms

Transfer of
energy to
gas atoms

High
energy gas
atoms
directed
towards
sample

Energy
transfer to
sample

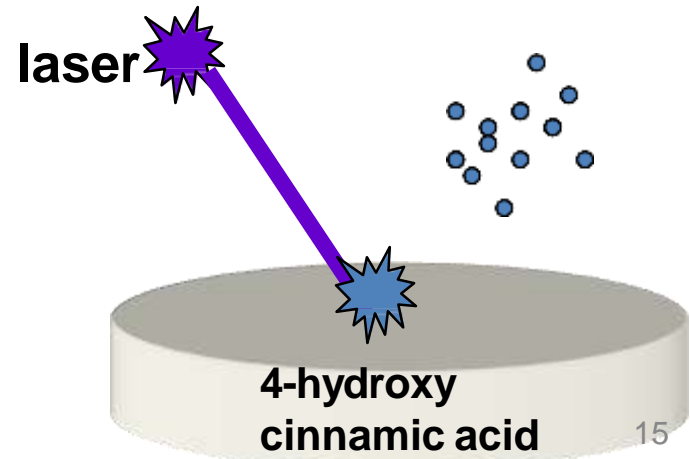
LASER DESORPTION TECHNIQUE:

Eg: **MALDI**(Matrix Assisted Laser Desorption Ionization)

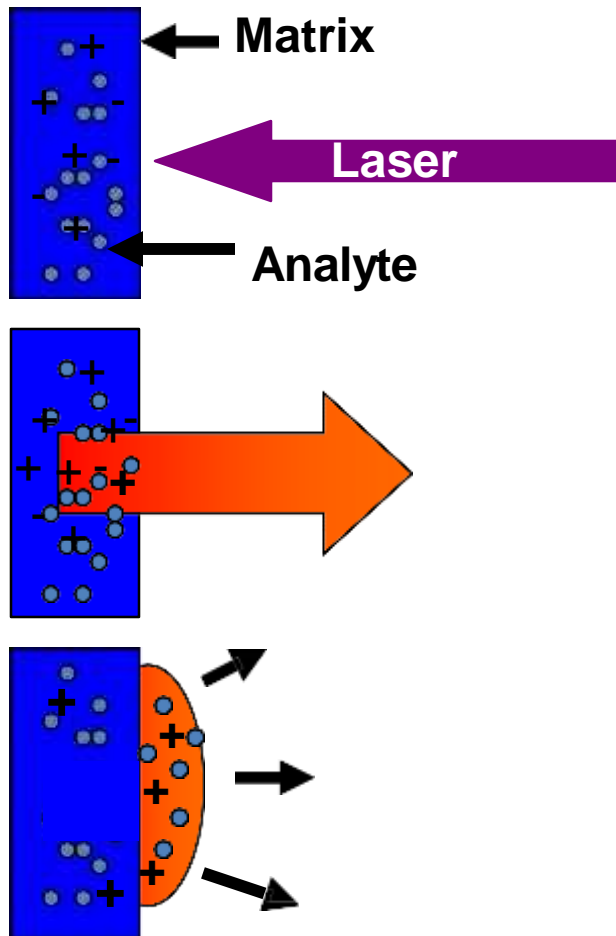
- TOF (Time Of Flight) analyzer

Advantages :

- ✓ No direct contact of sample and electron
- ✓ Easy to operate



Contd.....



Laser is bombarded with Matrix containing the sample

Energy transfer to sample

Expansion of matrix

MASS ANALYSERS

+ve ions at the end of chamber



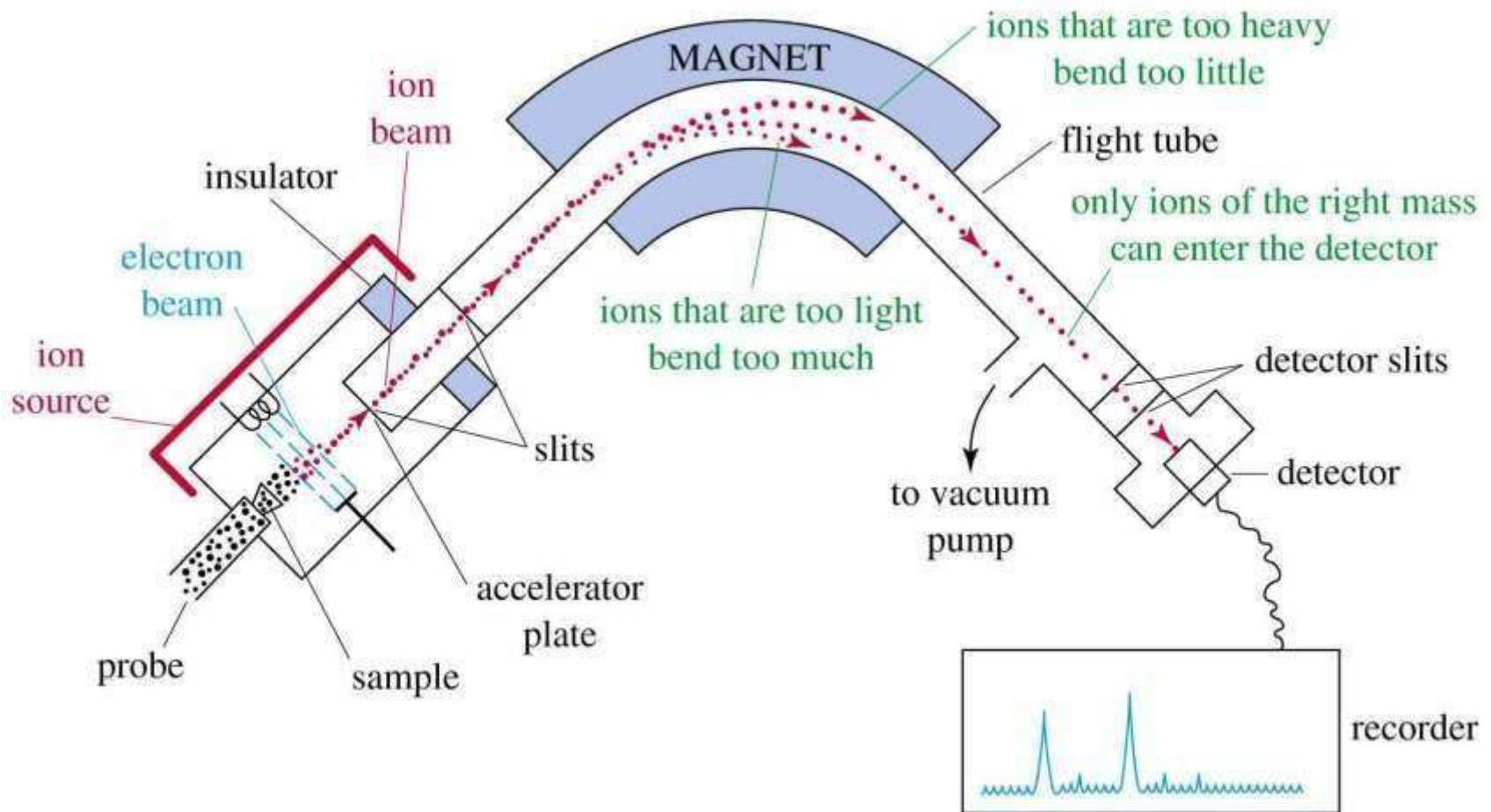
Bombarded with accelerating potential (2-8kV)



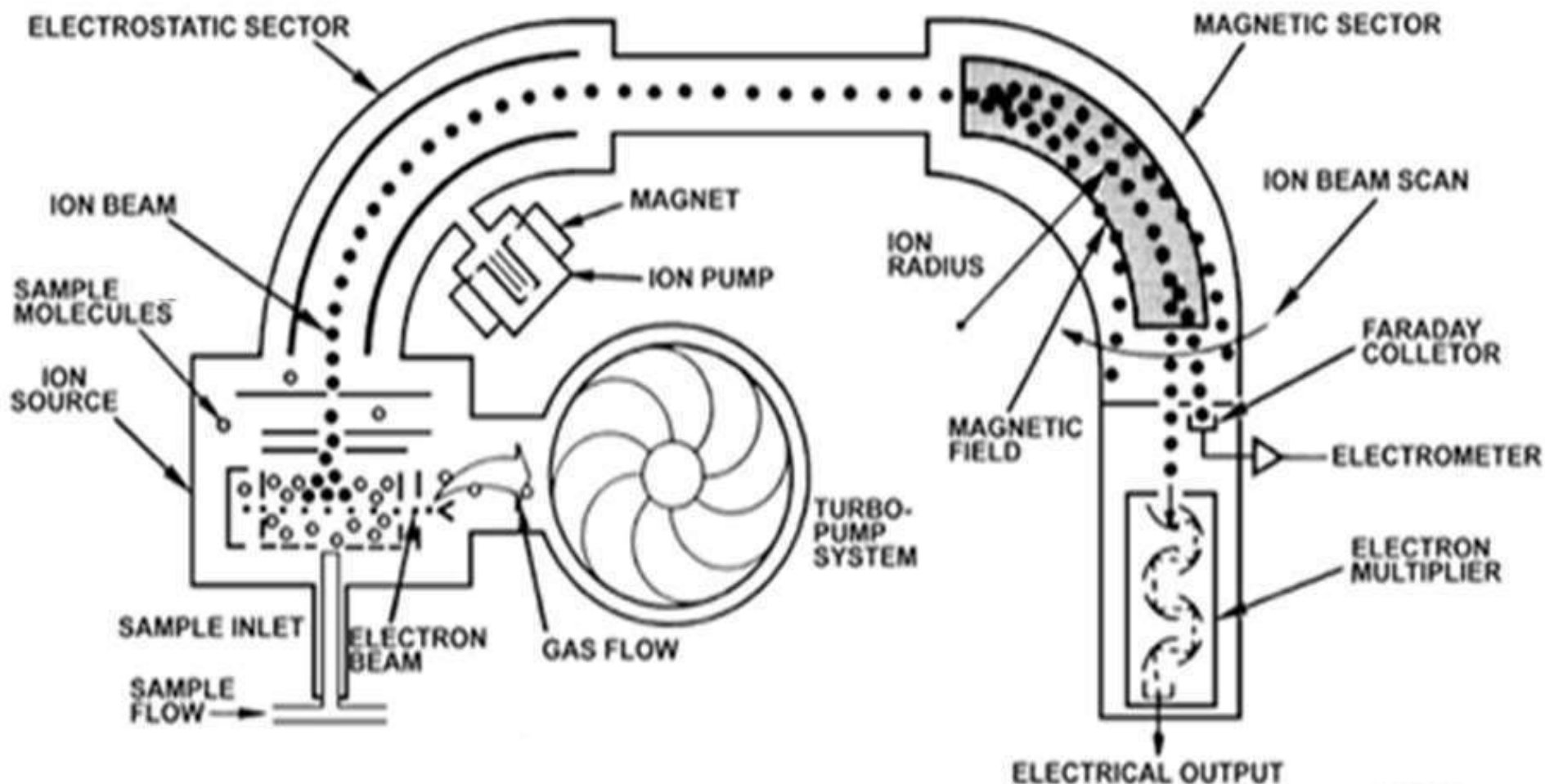
Deflection in magnetic field according to velocity & m/e

Analyzers are used to deflect ions based upon m/e ratio.

Single focusing magnetic analyser



Double focusing magnetic analyser



0-042-02

Single focusing

Magnetic field



Ions of same mass number are separated



Less sensitivity(10^{-1})

Double focusing

Magnetic field & electric field



Ions of same mass no. & same velocity are separated

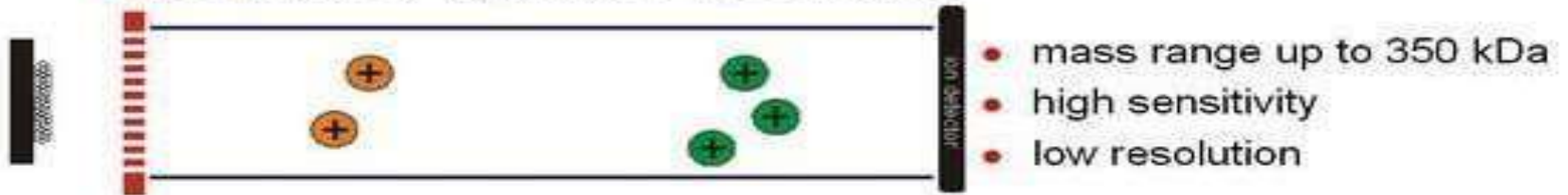


Highly sensitive(10^{-12})

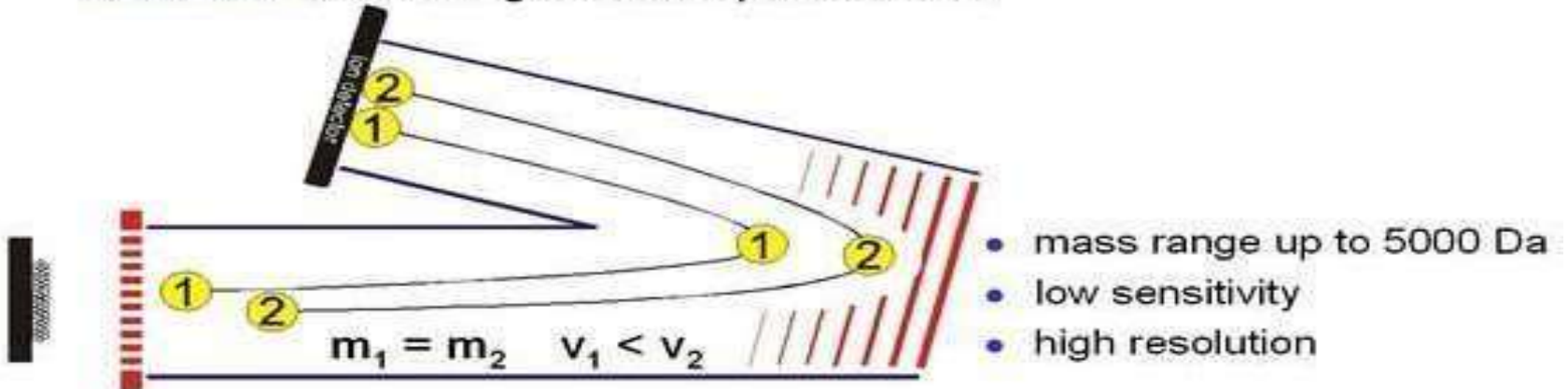
Time Of Flight(TOF)

Linear and reflector TOF MS

Linear time-of-flight mass spectrometer



Reflector time-of-flight mass spectrometer



DETECTORS



Least sensitive.
Cup will capture the ion.

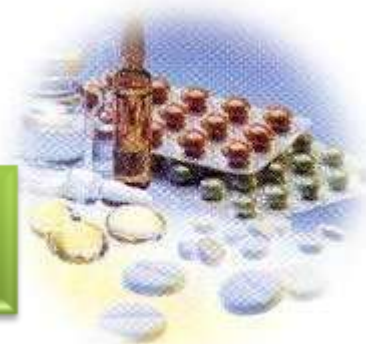


sensitive.
1000 times greater than faraday cup.



Most sensitive.
Used for high resolution.

APPLICATIONS



Molecular weight, formula & elemental composition determination

Identification of an unknown compound

Distinction between cis & trans- isomers

Identification of fragmentation pattern

Impurity detection

As LC-MS in analysis of proteins



Conclusion

- Mass spectrometry is used for the study of charged molecules and fragment ions. Thus it is useful for the identification of an unknown compound.

