

## 1/ Nuclear spectroscopy:

Many nuclear reactions produce radioactive products.

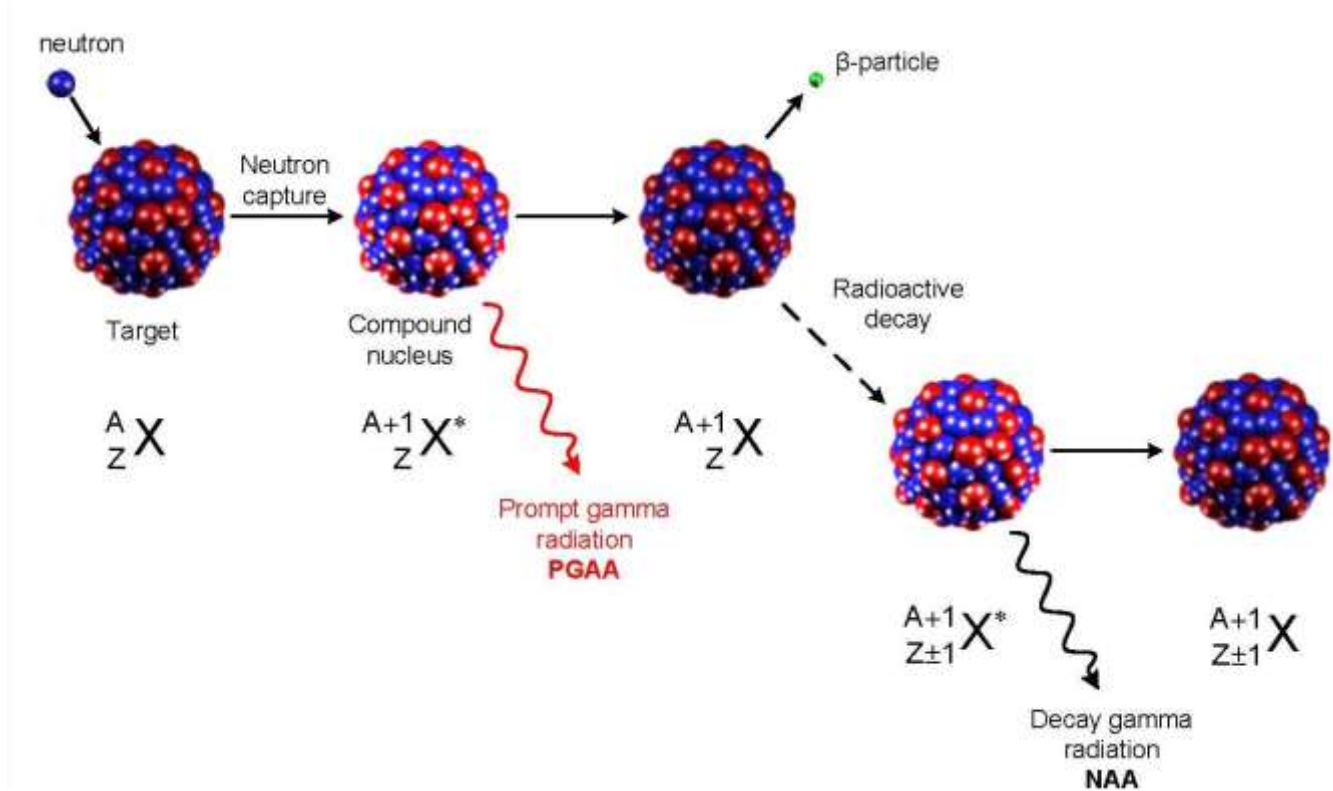
The most common of these reactions involve neutrons:



## 2/ Neutron Activation Analyses:

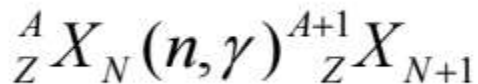
**NAA** is a physical technique that is based on nuclear reactions. The sample becomes radioactive when neutrons react with the nuclei of the elements' atoms. Radioisotopes are formed and subsequently decay by emitting gamma rays that are unique in half-life and energy (identification). Gamma-ray intensity is proportional to the element content in the sample.

Neutron activation analysis (NAA) is a nuclear process used for determining the concentrations of elements in a vast amount of materials. NAA relies on excitation by neutrons so that the treated sample emits gamma-rays. It allows the precise identification and quantification of the elements, above all of the trace elements in the sample. NAA has applications in chemistry but also in other research fields, such as geology, archeology, medicine, environmental monitoring and even in the forensic science.



Most common type of nuclear reaction for NAA

This produces isotopes through  $(n, \gamma)$  nuclear reactions in accordance with:



The  $\gamma$  energy is specific for the nucleus with a specific decay rate, and can be measured. If nuclide produced is radioactive, nuclear transformations will follow a 1-order kinetic reaction according to:

$$D_t = D_0 e^{-\lambda t}$$

Where:

$D_0$  = disintegration during irradiation termination

$D_t$  = disintegration after a time  $t$

$\lambda$  = disintegration constant

The disintegration constants are as we know, characteristic for each nuclide. Disintegration,  $D$ , is proportional to the number of radionuclides,  $N$ , giving:

$$D = \lambda N$$

where  $\lambda$  = disintegration constant,  $\ln 2/t_{1/2}$ ,

$t_{1/2}$  = the physical half-life,

$N$  = number of radionuclides.

### Detection of radionuclides:

The irradiated material is now radioactive and can be measured using conventional radiochemical methods i.e. a Ge-detector.

A certain amount of irradiated material is measured over a certain time, depending on the amount of radioactive material in the sample. Instrument background and blank samples measured in advance, and energy calibration and efficiency calibration of the respective geometries are added.

Disintegration measured in Becquerel (Bq), is used for quantitative detection. Decay of the radionuclides produced in an (n,  $\gamma$ ) reactions by irradiation finally given by:

$$D_T = \sigma \Phi N_T (1 - e^{-\lambda T})$$

Where:

$N_T$  = number of stable nuclides that are irradiated,

$\sigma$  = reaction probability (capture cross, 1barn =  $10^{-24}$  cm<sup>2</sup>),

$\Phi$  = neutron flux (number of neutrons sec<sup>-1</sup>cm<sup>-2</sup>) and

T = irradiation time.

After waiting ten from irradiation end disintegration given by:

$$D_t = \sigma \Phi N_T (1 - e^{-\lambda T}) e^{-\lambda t}$$

$$N_T = (m / M) X N_A$$

where

m = the amount of irradiated sample

M = the molar mass of the substance,

X = frequency of the irradiated isotope,

$N_A$  = Avogadro's number

#### a- Neutron sources :

##### a-1- Radio isotopic neutron sources:

- Two-component sources based on ( $\alpha$ ,n) reactions:  $\alpha$  decay from <sup>239</sup>Pu, <sup>241</sup>Am, <sup>210</sup>Po then <sup>9</sup>Be( $\alpha$ ,n)<sup>12</sup>C.
- Two-component sources based on ( $\gamma$ ,n) reactions: <sup>24</sup>Na, <sup>124</sup>Sb then <sup>9</sup>Be( $\gamma$ ,n) 2 <sup>4</sup>He.
- Spontaneous fission sources, e.g. <sup>252</sup>Cf.

*a-2- Neutron generators:*

- 2,4 MeV neutrons  $D(d, n) {}^3\text{He}$ .
- 14 MeV neutrons  $D(t, n) {}^4\text{He}$ .

*a-3- Cyclotrons:* d shot at Be target.

*a-4- Spallation neutron sources:* e.g. heavy elements such as W, Pb, U irradiated with high energy protons or other particles are spalled into two or more fragments and many neutrons are released.

*a-5- Neutron reactors:* mostly used, they allow for determinations using different neutron reactions and reactor neutron spectrum.

**b- Classification of NAA:***b-1- according to chemistry involved:*

- **Instrumental NAA (INAA):** no chemical treatment of sample is involved.
- **Radiochemical NAA (RNAA):** chemical separations are done after irradiation to remove interferences or to concentrate the radionuclide of interest.
- **Chemical NAA (CNAA):** pre-irradiation chemical separations are employed.
- **Molecular Activation Analysis (MAA):** specific molecular components are determined.

*b-2- according to energy of incoming neutrons:*

- **Thermal NAA (TNAA):** thermal  $n$ : their velocity is low ( $\approx 2200$  m/s) with mean energy of about 0.04 eV.
- **Epithermal NAA (ENAA):** epithermal  $n$ : their energy is in the range of 0.1 to 1 eV. This type of NAA usually comprise also resonance  $n$ , that are in the range of 1 eV to 1 KeV.
- **Fast NAA (FNAA):** fast  $n$ : their energy is  $>0.5$  MeV.
- **14-MeV INAA:** a form of FNAA based on reactions with 14-MeV neutrons that are produced by neutron generators.

**3/ Nuclear Magnetic Resonance (NMR):**

All atomic nuclei can be characterized by a nuclear spin quantum number  $S$ .

$S$  can be  $\geq 0$  and any multiple of  $\frac{1}{2}$ .

Nuclei with  $S = 0$  do not possess nuclear spin and consequently are termed 'NMR silent'.

Direct observation of the H's and C's of a molecules  
Nuclei are positively charged and spin on an axis;  
they create a tiny magnetic field.

Not all nuclei are suitable for NMR.

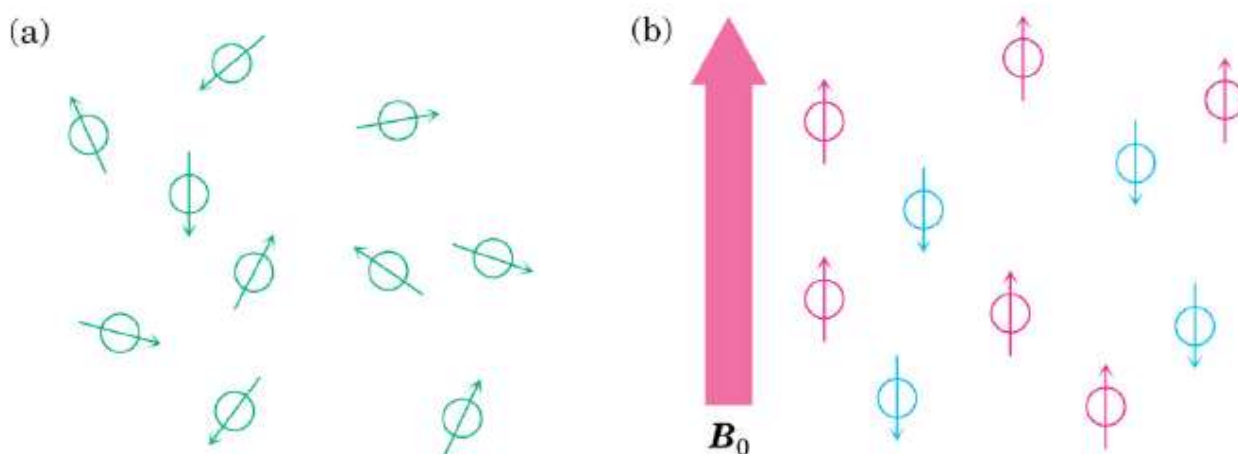
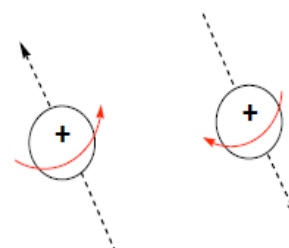
$^1\text{H}$  and  $^{13}\text{C}$  are the most important NMR active  
nuclei in organic chemistry.

Natural Abundance:

$^1\text{H}$  99.9%,  $^{13}\text{C}$  1.1%,  $^{12}\text{C}$  98.9% (not NMR active).

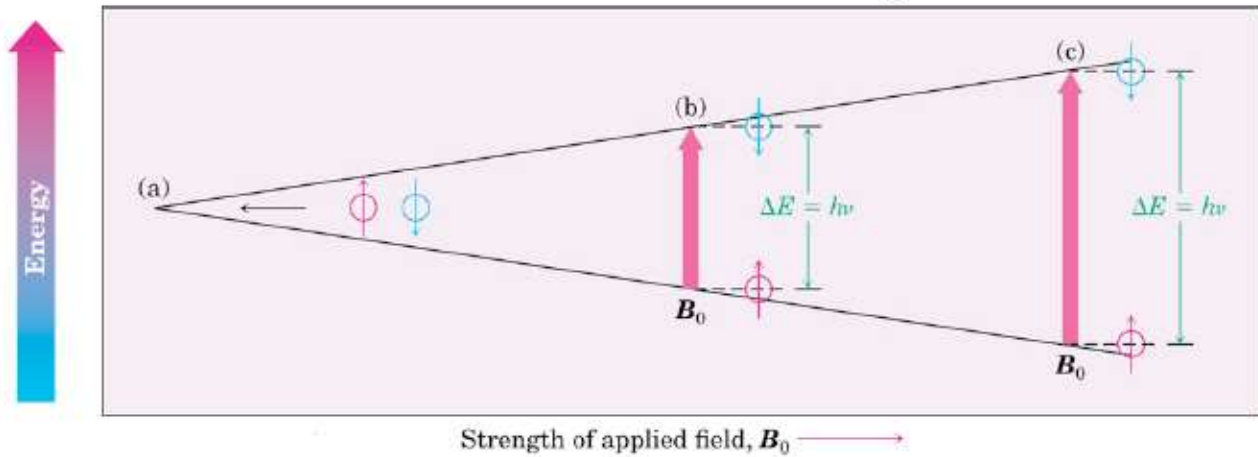
(a) Normally the nuclear magnetic fields are randomly oriented.

(b) When placed in an external magnetic field ( $\mathbf{B}_0$ ), the nuclear magnetic field  
can either be aligned with the external magnetic or oppose the external  
magnetic field.



The energy difference between aligned and opposed to the external magnetic  
field ( $B_0$ ) is generally small and is dependant upon  $B_0$ .

Applied EM radiation (radio waves) causes the spin to flip and the nuclei are  
said to be in **resonance** with  $B_0$ .



All nuclei with  $S \neq 0$  possess spin, charge, and angular momentum  $P$ , resulting in a nuclear magnetic moment  $\mu$ .

$\mu = \gamma \cdot P$  Where  $\gamma$  is the magnetogyric ratio of the nucleus.

$$\Delta E = h \nu, \quad \Delta E = \frac{\gamma B_0 h}{2\pi}$$

$B_0$  = external magnetic field strength

$\gamma$  = gyromagnetic ratio

$^1\text{H} = 26,752$

$^{13}\text{C} = 6.7$

**NMR Active Nuclei:** nuclear spin quantum number ( $S$ )

Number of spin states =  $2S + 1$  (number of possible energy levels)

Even mass nuclei that have even number of neutrons have  $S = 0$  (NMR inactive)

Even mass nuclei that have odd number of neutrons have an integer spin quantum number ( $S = 1, 2, 3, \text{etc}$ )

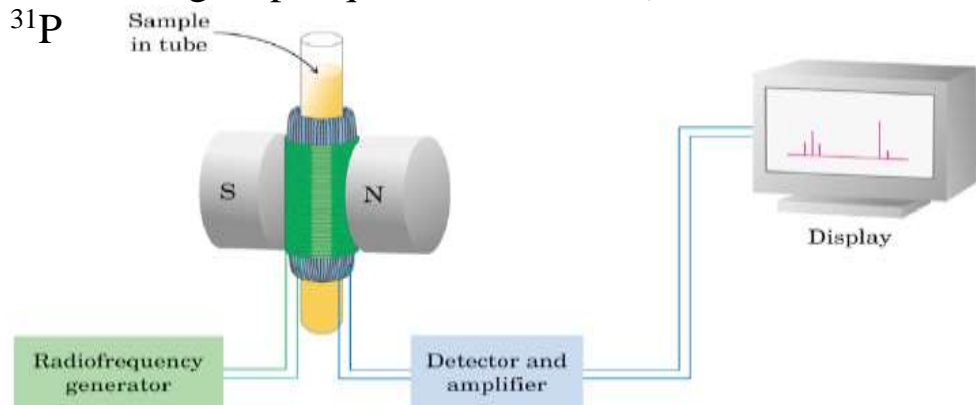
Odd mass nuclei have half-integer spin quantum number ( $S = 1/2, 3/2, 5/2, \text{etc}$ )

$S = 1/2$ :  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$

$S = 1$ :  $^2\text{H}$ ,  $^{14}\text{N}$

$S = 3/2$ :  $^{15}\text{N}$

$S = 0$ :  $^{12}\text{C}$ ,  $^{16}\text{O}$



Continuous wave (CW) NMR

Pulsed (FT) NMR