Glycosides

Physical Properties and Extraction

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Physical Characters

- Solids either amorphous or crystalline, Colorless
  - flavonoids - yellow,
  - anthraquinone - red or orange.
- Non volatile.
- Usually bitter in taste.
- Soluble in water and polar organic solvents.
- Reduce Fehling’s solutions only after hydrolysis.
- Give positive reaction with Molisch's and Fehling's solution test (after hydrolysis)

Solubility

- glycosides are water soluble compounds and insoluble in the organic solvents.
  - Glycone part: water soluble, insoluble in the organic solvents.
  - Aglycone part: water insoluble, soluble in the organic solvents
- Some glycosides are soluble in alcohol.

Stability of Glycosides

Effect of acid hydrolysis:

- **Acids split sugars from the aglycones.**
- **The acetal linkage is more readily cleaved than the linkage between the individual sugars of the sugar chain.**
- **C-glycosides are resistant to acid hydrolysis.**

Effect of alkaline hydrolysis

A- Strong alkalis:

- Hydrolysis of ester groups
- Opening of lactone rings
  - e.g. Cardiac glycosides

B- Mild alkalis:

- Hydrolysis of ester groups
  - e.g. Lanatoside A to Purpurea A
- Opening of lactone rings
  - e.g. Cardiac glycosides

Enzymatic hydrolysis:

- Split the sugars stepwise starting from the terminal sugars.
- All plants producing glycosides have enzyme that can hydrolyze these glycosides.
- Enzymes are specific for the type of glycosidic linkages:
    - Emulsin can hydrolyze β – glycosides
    - Invertase can hydrolyze α- glycosides
    - Myrosin can hydrolyze s-glycosides

Extraction and Isolation

- **Because of the wide range of physical and chemical properties of glycosides and other constituents associated with them, no common general method for their isolation is recommended.**
- **Water, methanol, water-ethanol and ethanol are the most common solvents for extraction of glycosides.**

Precautions before extraction

Deactivation of enzymes:

- **Drying** for 15-30 min at **100˚C** followed by slow drying at a low temperature.
- Dipping the fresh material into **boiling water** or **boiling alcohol** for 10-20 min.
- **Boiling** the fresh plant material with **acetone**.
- Carrying out the extraction at very low temp.
- **Freeze-drying** of the plant material **before extraction** (lyophilization).
- Carrying the extraction in the presence of $(NH_4)_2SO_4$.

Maintenance of neutral conditions:

- **Neutral pH should be assured before and during extraction because:**
- **Acidity may result in hydrolysis. This is overcome by addition of $CaCO_3$.**
- **Mild alkalinity may sometimes produce racemization.**

Defatting of fat-rich organs (e.g. seeds) before extraction:

- **High amounts of lipoids hinder glycoside extraction.**
- **Defatting is usually carried with petroleum ether**

SEPARATION OF GLYCONE & AGLYCONE

Glycosides $\xrightarrow[\text{dil. HCl}]{\text{Hydrolysis}}$ **Glycone + Aglycone + HCl**

$\xrightarrow[\text{Using alkali}]{\text{Neutralization by}}$ **Glycone +Aglycone +salt+H_2O** $\xrightarrow{\text{Filtration}}$

H_2O+ Glycone +Aglycone $\xrightarrow{\text{chloroform}}$

(Glycone in H_2O) + (Aglycone in chloroform)

We can separate them by using separatory funnel

The best solvent to extract aglycone is Ethyl acetate because it is:

- **immiscible in water**
- **always presents in the upper layer**

- **Note: Alcohol and acetone are water miscible solvents, so we can't use them as organic solvents for aglycone separation**