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SAMPLE: Ascorbic Acid

BEHAVIOUR OF COMPOUND DURING HEATING AND BURNING (*describe what you should see during the heating of your sample in burner and choose one of possibility*):

turning black, carbonization- EVERY TIME!

- increasing of volume
- melting
- releasing vapours that are flammable
- sublimation

An organic compound will be burnt without a rest (it can leave a black coating on inner sides of a fusion tube, because of insufficient access of the air).

-----ORGANIC-----/INORGANIC/ORGANIC-INORGANIC COMPOUND

ELEMENTARY ANALYSIS (*write down the reactions of tests you should do and mark which of them should be positive*):

HALOGENS (HALIDES)

Acidify a portion (5 mL) of the filtrate with dilute nitric acid, and if nitrogen and/or sulphur are present, boil for 1 - 2 minutes. Cool and add aqueous silver nitrate.

Formation of a heavy, white, yellowish or yellow precipitate of silver halide indicates halogen.

Should be negative

1. NITROGEN (CYANIDES)

To a portion (5 mL) of the filtrate add a few drops of ferrous sulphate solution and a few drops of ferric chloride solution. Boil the mixture for half a minute, cool and acidify by adding dilute hydrochloric acid drop wise. Formation of a bluish-green precipitate (Prussian blue) or a blue solution indicates that the original substance contains nitrogen. If no precipitate appears, allow to stand for 15 minutes, filter and inspect filter paper.

C_{org.} + N_{org.} CN-

6 CN⁻ + Fe²⁺ [Fe(CN)₆]⁴⁻

[Fe(CN)₆]⁴⁻ + Fe³⁺ {Fe^{III}[Fe^{II}(CN)₆]}-

Should be negative

2. SULPHUR (SULPHIDE)

To the cold filtrate (5 mL) add a few drops of lead acetate solution.

Production of a black solution or a black precipitate indicates that the original substance contains sulphur.

Sorg. S²⁻

S²⁻ + Pb²⁺ → PbS

Should be negative

No Sulphur no halides and no halogens present.

SOLUBILITY (*decide according to the information in Ph. Eur.*):

- **Freely soluble in water, sparingly soluble in ethanol**

pH of solution/suspension (*decide according to nature of your sample*):

- **2.1-2.6 solution**

REACTIONS FROM THE FLOWCHARTS (*write down your “flowcharts pathway”; describe results of your hypothetical analysis – reactions from the flowcharts you can find in material called “Identification of an unknown drug”*):

Identification of an organic compound containing C, H, O

It is soluble in water and is acidic .

Ascorbic acid or Citric acid or Tartaric acid

IDENTIFICATION REACTIONS (*from your monography choose the tests necessary for identification of your substance and describe them*):

First identification: B, C

Second identification: A, C, D

- A. Ultraviolet and visible absorption spectrophotometry**

Test solution: Dissolve 0,10g in water R and dilute immediately to 100.0ml with the same solvent. Add 1.0ml of the solution to 10ml of a 10.3g/L solution of HCL R and dilute to 100.0ml with water R.

B. Infrared absorption spectrophotometry

C. pH (2.2.3): 2.1 to 2.6 for solution S

Solution S : dissolve 1,0g in carbon dioxide free water R and dilute to 20 ml with the same solvent.

D. To 1ml of solution S add 0.2ml of dilute nitric acid R and 0.2ml of silver nitrate solution R2. A grey precipitate is formed.