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SAMPLE: Glucose monohydrate

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*):

An organic compound will be burnt without a rest it can leave a black coating on inner sides of a fusion tube.

ORGANIC/INORGANIC/ORGANIC-INORGANIC COMPOUND

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

TESTS DESCRIBED BELOW:

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.

Org. + Norg. CN⁻

$6 \text{CN}^- + \text{Fe}^{2+} \rightarrow [\text{Fe}(\text{CN})_6]^{4-}$

$[\text{Fe}(\text{CN})_6]^{4-} + \text{Fe}^{3+} \rightarrow \{\text{Fe}^{\text{III}}[\text{Fe}^{\text{II}}(\text{CN})_6]\}^-$

If the organic compound contains both nitrogen and sulphur, we must modify the procedure:

Add a few drops of dilute sodium hydroxide to 5 mL of the filtrate and then add ferrous sulphate solution drop wise until the precipitate stops to form. Boil the mixture, filter it, acidify the filtrate by adding dilute hydrochloric acid and finally add ferric chloride solution.

A blue precipitate forms.

Should be positive

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⁻

$\text{S}^{2-} + \text{Pb}^{2+} \rightarrow \text{PbS}$

Should be negative

3. 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.

Don't throw the precipitate away!

Should be negative

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

- The sample should dissolve totally in water

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

- The pH will be neutral -

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"*):

- Organic compound and in glucose we have C,H and O so I go to flowchart 3
 - o It's soluble in water
 - o pH of the solution is neutral
 - I do reaction with ferric chloride solution turn negative
 - So I do the reaction with Fehlin's reagent turn positive
 - o We can conclude we have Glucose or Fructose or Lactose

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

Specific optical rotation:

Dissolve 10g in 80mL of water, add 0,2mL of dilute ammonia R1, allow to stand for 30 minute and dilute to 100,0mL with water

The specific optical rotation is + 52.5 to + 53.3, calculated with reference to the anhydrous substance.

Examine the chromatograms obtained in the assay

The principal peak in the chromatogram obtained with the solution is similar in retention time and size to the principal peak in the chromatogram obtained with reference solution a

We have to do thin layer chromatography

Test solution. Dissolve 10 mg of the substance to be examined in a mixture of 2 volumes of water R and 3 volumes of methanol R and dilute to 20 ml with the same mixture of solvents. Reference solution (a). Dissolve 10 mg of glucose CRS in a mixture of 2 volumes of water R and 3 volumes of methanol R and dilute to 20 ml with the same mixture of solvents. Reference solution (b). Dissolve 10

mg each of fructose CRS, glucose CRS, lactose CRS and sucrose CRS in a mixture of 2 volumes of water R and 3 volumes of methanol R and dilute to 20 ml with the same mixture of solvents. Apply separately to the plate 2 µl of each solution and thoroughly dry the starting points. Develop over a path of 15 cm using a mixture of 10 volumes of water R, 15 volumes of methanol R, 25 volumes of anhydrous acetic acid R and 50 volumes of ethylene chloride R. The solvents should be measured accurately since a slight excess of water produces cloudiness. Dry the plate in a current of Reaction warm air. Repeat the development immediately, after renewing the mobile phase. Dry the plate in a current of warm air and spray evenly with a solution of 0.5 g of thymol R in a mixture of 5 ml of sulphuric acid R and 95 ml of alcohol R. Heat at 130 °C for 10 min.

The principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with reference solution (a). The test is not valid unless the chromatogram obtained with reference solution (b) shows 4 clearly separated spots.

Identification test :

We have to dissolve 0,1g in 10mL of water R, add 3mL of cuprt-tartaric solution R and heat.

A red precipitate should formed

Water :

7.0 per cent to 9.5 per cent, determined on 0.50 g by the semi-micro determination of water.