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SAMPLE: Salicylic 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)***:**

ca 0.01 g of your compound into a fusion tube. Take the fusion tube by tongs and bring it slowly near the flame of a burner. Watch all consecutive changes of the appearance, colour and state. Finally put the fusion tube directly into the flame and anneal it well.

Since my sample is an <u>organic compound</u>

Organic compounds- possible changes during heating and burning:

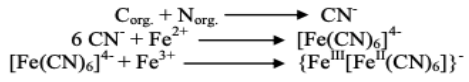
- **turning black, carbonization- EVERY TIME!**
- **increasing of volume**
- **melting**
- **releasing vapours that are flammable**
- **sublimation**

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

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

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 (Prussianblue) 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

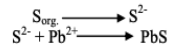


If the organic compound contains both nitrogen and sulphur, we must modify the procedure: Add a few drops of dilute sodium hydroxide to 5 mL of the filtrate and then add ferrous sulphate solution drop wise until the precipitate stops to form. Boil the mixture, filter it, acidify the filtrate by adding dilute hydrochloric acid and finally add ferric chloride solution. A blue precipitate forms.

our sample gives positive reaction.

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.



our sample gives negative reaction

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

   our sample gives negative reaction

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

slightly soluble in water and it's freely soluble in ethanol (96%)

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

acidic , The PH is between 3-4

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")***:**

So my sample is salicylic acid which is an organic compound and the formula is C7H6O3 so this sample contains C,H,O so we go with flow chart 3 and from the Ph. Eur. we can say that this sample

is almost insoluble in water but can be freely soluble in ethanol and we have an acidic ph which is roughly between 3-4 so based on the given flow chart we can identify that we have Salicylic acid

and since salicylic acid contains a phenolic –OH group gives positive reaction to Ferric chloride which the reaction is described as below :

Dissolve ca 0.1 g of the compound in 10 mL of distilled water. Take 5 ml of the solution and add 1-2 drops of ferric chloride solution. The solution turns blue- violet.

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

First identification : A,B

Second Identification: A,C

A. **Melting point : 158 C to 161 C**
B. **Infrared absorption spectrophotometry**
C. **Dissolve about 30mg in 5ml of 0.05 M sodium hydroxide, neutralise if necessary and dilute to 20 ml with water R. 1ml of the solution gives reaction (a) which is "** *to 1ml of the prescribed solution add 0.5ml of ferric chloride solution R1. A violet colour is produced that persists after the addition of 0.1 ml of acetic acid* **R'' of salicylates**