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SAMPLE: Resorcinol (C6H602)

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)***:**

Our sample is Resorcinol and it is an organic compound. During the heating in burner we can see carbonization take place which is mean that it will turn black. Also sublimation it might take place while releasing vapours that are flammable. Volume of Resorcinol might increasing during melting time. Also an organic compound like Resorcinol will be burnt without a rest.

ORGANIC/INORGANIC/ORGANIC-INORGANIC COMPOUND

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

The elements commonly occurring along with carbon and hydrogen in organic compounds are oxygen, nitrogen, sulphur, chlorine, bromine and iodine. The detection of these elements depends upon converting them to water soluble ionic compounds and the application of specific tests. There can be performed an oxidative mineralisation (with CuO) or reductive mineralisation (with Na and Mg).

Lassaigne's test after reductive mineralisation.

C, H, O, N, S, X -> X- CN- S(2-) CNS-

PROCEDURE:

Place a little of the compound (50 mg or 2 - 3 drops) into a fusion tube. Add a reaction mixture (magnesium powder + potassium carbonate) along the fusion tube length so that a gap for hot air to escape remains there.

Heat the fusion tube gently at first by flame of a gas burner. Then heat up the reaction mixture. When charring begins, heat the bottom of the tube to dull redness (but avoid melting of the glass!).

Finally plunge the tube, while still hot, into a clean dish test tube half-filled with cold distilled water kept in a test tube stand. The fusion tube breaks and the content is released into the water. Filter it, add distilled water to 15 mL and divide the filtrate into 3 parts.

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.

Corg. + Norg.-> CN-

6 CN- + Fe2+ ->[Fe(CN)6]4-

[Fe(CN)6]4- + Fe3+-> {FeIII[FeII(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. So this test is 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. Test should be negative.

Sorg. -> S2-

S2- + Pb2+ -> PbS

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!

So also this test is negative because we know from our pharmacopeia to acidify our solution needs to 10ml of solution S add 0.05ml of bromophenol blue solution. Not more than 0.05ml of 0.1M hydrochloric acid / 0.1 M sodium hydroxide is required to change the colour of the indicator.

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

Very soluble in water and in alcohol

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

Take a watch glass and put a piece of an indicator on it. Then use a clean glass rod to transport a drop of the solution Resorcinol of on the indicator paper. Compare its colour with the scale on the package of indicator papers and determine pH.

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")***:**

Flowcharts pathway:

Unknown compound (Resorcinol) => organic compounds=> elementary analysis=> C,H,O => Flowchart 3

So first step of our identification is solubility with water. Our sample is soluble in water and in alcohol.

Then we continue with pH of our solution which is neutral.

We can continue with the reaction with ferric chloride solution. In this reaction we will dissolve ca 0.1g of the compound in 10 ml of distilled water. Then we will take 5ml of the solution and add 1-2 drops of ferric chloride solution. At the end our solution we turn blue-violet.

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

Melting point: 109 to 112°C

Dissolve 0.1g in 1 ml of water R, add 1 ml of strong sodium hydroxide solution R and 0.1ml of chloroform R, heat and allow to cool. An intense, deep- red colour develops which becomes pale yellow on the addition of a slight excess of hydrochloric acid R.

Thoroughly mix about 10 mg with about 10mg of potassium hydrogen phthalate R, both finely powdered. Heat over a naked flame until an orange-yellow colour is obtained. Cool and add 1 ml of dilute sodium hydroxide solution R and 10ml of water R and shake to dissolve. The solution shows an intense green fluorescence.