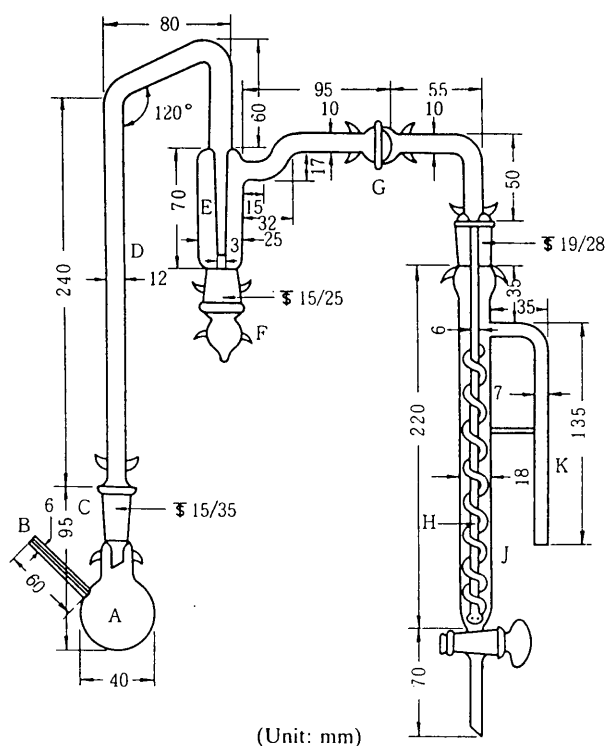


B. GENERAL TESTS

Methoxyl Determination

The Methoxyl Determination Test is designed to quantify methoxyl group, in which a sample is heated with hydriodic acid, the produced methyl iodide is oxidized with bromine, and the produced iodide acid is titrated with sodium thiosulfate solution.

Apparatus Use the apparatus illustrated in the figure below.



- A: Decomposition flask
- B: Gas-introducing tube
- C: Ground-glass joint
- D: Air condenser
- E: Gas scrubber
- F: Glass stopper
- G: Ball joint
- H: Gas duct
- J: Absorption tube
- K: Gas-exhaust tube

B. GENERAL TESTS

Preparation of Scrubbing Solution and Absorbing Solution

Rinsing solution (Scrubbing Solution) Weigh 1 g of red phosphorus, and suspend in 100 ml of water.

Absorbing solution Weigh 15 g of potassium acetate, and dissolve in 150 ml of an acetic acid-acetic anhydride mixture (9 : 1). Measure 145 ml of the solution, and add 5 ml of bromine. Prepare freshly before use.

Procedure Pour the rinsing solution into about a half height of gas scrubber E, and transfer about 20 ml of the absorbing solution into absorption tube J. Weigh accurately the sample corresponding to about 6.5 mg as methoxyl group (CH_3O : 31.03), transfer to decomposition flask A, and add boiling chips and about 6 ml of hydriodic acid. Moisten ground-glass joint C of A with 1 drop of hydriodic acid, and connect A to condenser D. Assemble the apparatus by connecting ball joint G, using a suitable silicone resin. Pass nitrogen or carbon dioxide through gas-introducing tube B, and adjust the flow rate so that bubbles appear in E at the rate of 2 bubbles per second, using a suitable pressure-regulating device. Place A in an oil bath, heat the flask until the temperature of the bath becomes 150°C after heating for 20 or 30 minutes, and boil for further 60 minutes. Remove the oil bath, allow to cool the flask under the gas stream, and separate G after cooling. Transfer the contents of J into a 500-ml Erlenmeyer flask with a ground-glass stopper containing 10 ml of sodium acetate solution (1 : 5), wash the tube with water several times, adding the washings to the flask, and finally dilute to about 200 ml with water. Add dropwise formic acid with shaking until the red color of the bromine disappears, and add further 1 ml of formic acid. Then, add 3 g of potassium iodide and 15 ml of diluted sulfuric acid (1 : 20), stopper, shake gently, allow to stand for 5 minutes, and titrate the liberated iodine with 0.1 mol/l sodium thiosulfate (indicator: 1 ml of starch TS). Perform a blank test in the same manner, and make any necessary correction.

1 ml of 0.1 mol/l sodium thiosulfate = 0.5172 mg of CH_3O