

Appendix X: Determination of Water Content

Method and Procedure

(1) Toluene distillation method

- (a) **Preparation of test sample** – Prepare a suitable quantity of test sample by cutting (or by using other appropriate means) CMM sample into pieces of less than 3 mm in length or diameter. Flowers, seeds or fruits of length or diameter less than 3 mm, can be used directly for the examination.
- (b) **Reagent** – Toluene used in this method should be saturated with water and distilled.
- (c) **Apparatus** – The apparatus (Fig. 1) consists of a 500-mL round-bottomed flask (A); a graduated receiving tube (B); and a reflux condenser (C) of about 40 cm in length. The apparatus should be cleaned and dried in an oven before being used.
- (d) **Procedure** – Weigh accurately a quantity of the sample which is expected to give 2-4 mL of water, transfer to the flask and add about 200 mL of toluene into it. When necessary, add a few pieces of glass beads. After assembly of the apparatus, fill in the narrow part of the receiving tube with toluene through the condenser, then heat the flask gently by using an electric heater or other appropriate means. When the toluene begins to boil, adjust the temperature to allow the distillation proceed at a rate of 2 drops per second until the water has been completely distilled. Rinse the inside of the condenser with toluene. Continue the distillation for five more minutes, then remove the apparatus away from the heat and allow it to cool to room temperature. Disconnect the apparatus and dislodge any droplets of water that adhere to the wall of the receiving tube.

Allow the receiving tube to stand a while until the water and toluene are completely separated [Note]. Record the volume of water and calculate the percentage of water content in the weight of CMM sample.

Note: A small amount of methylene blue may be added to form a bluish aqueous layer to facilitate observation.

(2) Oven dried method

Place 2 to 5 g of powdered sample being examined in a flat weighing bottle, previously dried to constant weight, to form a smooth layer not exceeding 5 mm in thickness, or not exceeding 10 mm in thickness for

sample of loose texture and then weigh accurately. Dry in an oven at 100 to 105 °C for 5 h with the stopper of the bottle removed. Upon opening the oven, close the bottle promptly and allow it to cool in a desiccator for 30 min. Weigh accurately and dry it again under similar condition for 1 h, cool and weigh. Repeat the operation until the difference between two successive weighings is not more than 5 mg. Calculate the percentage of water content in the weight of CMM sample.

Limits – The water content in CMM samples should not be more than the percentage specified in the individual monograph.

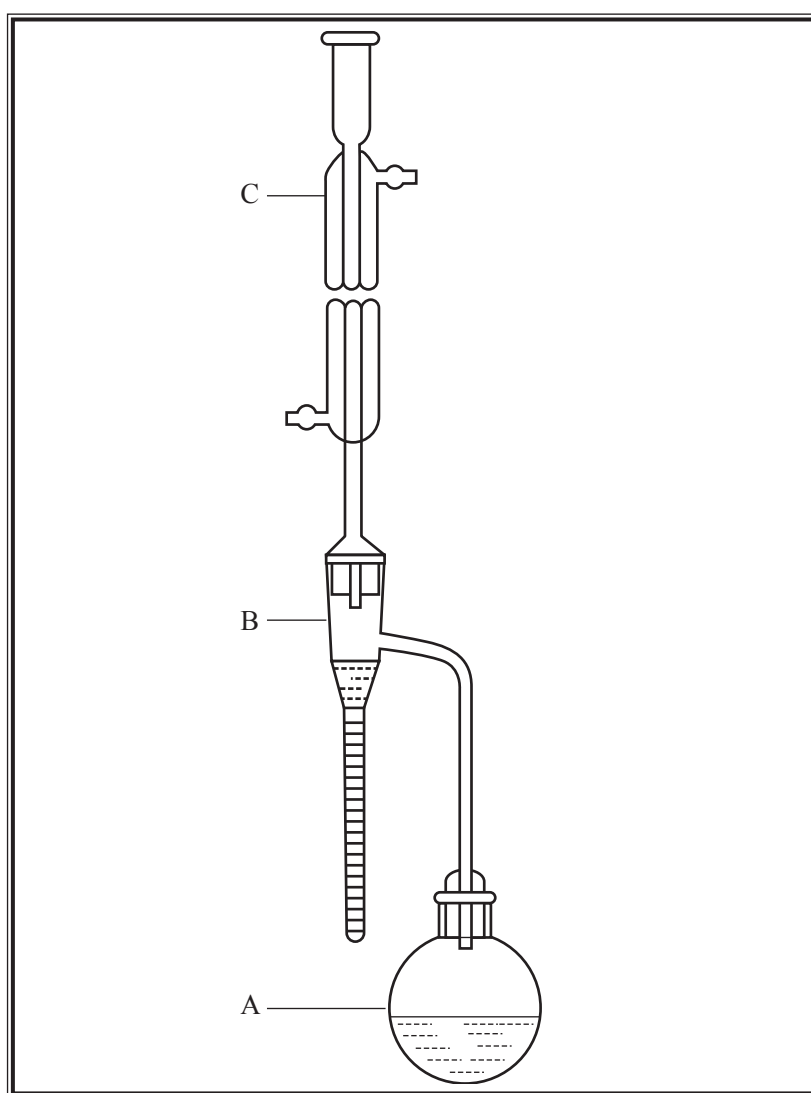


Figure 1 Apparatus for the determination of water content in CMM samples

- A. Round-bottomed flask
- B. Graduated receiving tube
- C. Reflux condenser