

Appendix XIII: Determination of Volatile Oil

Method – Specified apparatus is used to determine the volatile oil in CMM samples.

- (1) **Preparation of test sample** – Pulverize CMM sample, pass through No.2 or No.3 sieves and mix well, unless otherwise specified.
- (2) **Apparatus** – The apparatus (Fig. 1) consists of a 1000-mL (500-mL or 2000-mL) round-bottomed flask (A), a volatile oil determination tube (B) and a reflux condenser (C). All parts are connected via ground glass joints. The measuring tube of B is graduated in 0.1 mL. The apparatus should be cleaned before use and all parts of apparatus should be tightly connected to avoid the loss of volatile oil.

Note: The volatile oil determination tube should be set vertically. The connecting point between the side tube and the graduated tube is at a horizontal level.

(3) Procedure

- (a) **Method A** – This method is used to determine the volatile oils with relative density less than 1.0.

Take a quantity of the powdered sample which is expected to give 0.5-1.0 mL of volatile oil, weigh accurately to the nearest 0.01 g, and put into a round-bottomed flask. Add 300-500 mL of water (or appropriate amount) and a few glass beads, shake and mix well. Connect the round-bottomed flask to a volatile oil determination tube and then connect the volatile oil determination tube to a reflux condenser. Add water through the top of reflux condenser until the graduated tube of volatile oil determination tube is filled and overflows to the round-bottomed flask. Heat the flask gently until boiling by using an electric heating jacket or other appropriate means. Continue the gentle boiling for about 5 h until the volume of oil does not increase. Stop heating, allow it to stand for a while. Open the stopcock at the lower part of volatile oil determination tube and run off the water layer slowly until the oily layer is 5 mm above the zero mark. Allow to stand for at least 1 h, open the stopcock again, run off the remaining water layer carefully until the oily layer is just on the zero mark. Record the volume of oil in the graduated tube of volatile oil determination tube and calculate the percentage of volatile oil in CMM sample.

- (b) **Method B** – This method is used to determine the volatile oils with relative density more than 1.0.

Add 300 mL of water and a few pieces of glass beads into a round-bottomed flask. Connect the round-bottomed flask to volatile oil determination tube. Add water through the top of volatile oil determination tube until the graduated tube is filled and overflows to the round-bottomed

flask. Add 1 mL of xylene by using a pipette and then connect the reflux condenser to volatile oil determination tube. Heat the flask until boiling, continue the heating to allow the distillation proceed at a rate that will keep the middle part of the condenser cold. Stop heating after 30 min, and allow it to stand for at least 15 min. Record the volume of xylene in the graduated tube of volatile oil determination tube.

Carry out the procedure as described in Method A beginning at the words “Take a quantity of the powdered sample”. Subtract the volume of xylene previously observed from the volume of oily layer, the difference in volume is taken to be the content of volatile oil, calculate the percentage of volatile oil in CMM sample.

Limits – The CMM samples contain not less than the percentage of volatile oil specified in the individual monograph.

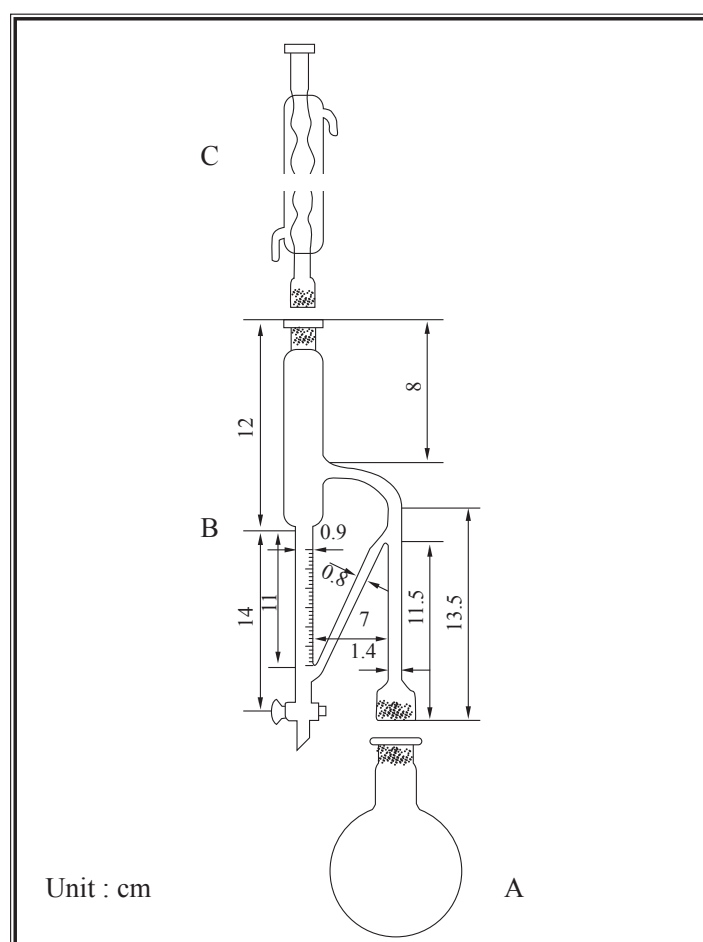


Figure 1 Apparatus for the determination of volatile oil in CMM samples

- A. Round-bottomed flask
- B. Volatile oil determination tube
- C. Reflux condenser