Appendix XI: Determination of Extractives

Method and Procedure –

(1) **Determination of water-soluble extractives** – Pulverize CMM sample, pass through a No.2 sieve and mix well.

(a) **Cold extraction method** – Place 4.0 g of the powdered sample, accurately weighed, in a 250-300 mL conical flask with a stopper. Accurately add 100 mL of water, insert the stopper and extract for 24 h. Shake frequently during the first 6 h, then allow to stand for 18 h. Filter rapidly through a dry filter. Accurately transfer 20 mL of the filtrate to an evaporating dish, previously dried to constant weight, and evaporate to almost dryness on a water bath, then dry at 105°C for 3 h. Cool in a desiccator for 30 min, and then weigh immediately and accurately. Calculate the percentage of water-soluble extractives with reference to the dried CMM sample.

(b) **Hot extraction method** – Place 2.0-4.0 g of the powdered sample, accurately weighed, in a 100-250 mL conical flask with a stopper. Accurately add 50-100 mL of water, insert the stopper and weigh. Allow to stand for 1 h. Attach a reflux condenser to the flask and boil gently for 1 h, then cool to room temperature and weigh, readjust to the original weight with water. Shake and filter through a dry filter. Accurately transfer 25 mL of the filtrate to an evaporating dish, previously dried to constant weight, and evaporate to dryness on a water bath, then dry at 105°C for 3 h. Cool in a desiccator for 30 min, and then weigh immediately and accurately. Calculate the percentage of water-soluble extractives with reference to the dried CMM sample.

(2) **Determination of ethanol-soluble extractives** –

(a) **Cold extraction method** – Proceed as in section 1(a) except by using ethanol (70%) in lieu of water as solvent.

(b) **Hot extraction method** – Proceed as in section 1(b) except by using ethanol (70%) in lieu of water as solvent.

Limits – The amount of water-soluble extractives and ethanol-soluble extractives in CMM samples should not be less than the percentages specified in the individual monograph.