Appendix IX: Determination of Ash

Method and Procedure –

(1) **Total ash** –

(a) Pulverize CMM sample, pass through a No.2 sieve and mix well. Accurately weigh 2-3 g (3-5 g for the determination of acid-insoluble ash) of the powdered sample in a tared crucible (to the nearest 0.01 g). Ignite the sample gently until completely carbonized, keep it from burning, then gradually increase the temperature to 500-600°C. Continue the ignition until a constant weight of carbon-free ash is obtained. Calculate the percentage of total ash in the weight of CMM sample.

(b) If a carbon-free ash cannot be obtained in this way, cool the crucible, and moisten the residue with hot water or 2 mL of aqueous ammonium nitrate solution (10%, v/v), then dry the residue on a water bath. Ignite the residue again as directed above until a carbon-free ash is obtained.

(2) **Acid-insoluble ash** –

To a crucible containing the total ash, add 10 mL of dilute hydrochloric acid (10%, v/v), cover with a watch glass and gently heat for 10 min on a water bath. Rinse the watch glass with 5 mL of hot water and add the rinsing to the crucible. Transfer the insoluble matter and rinse the remaining residues from the crucible onto an ashless filter paper, wash with hot water until the filtrate is free of chlorides. Transfer the ashless filter paper containing the insoluble matter to the original crucible, dry and ignite to constant weight. Calculate the percentage of acid-insoluble ash in the weight of CMM sample.

Limits – The amount of total ash and acid-insoluble ash in CMM samples should not be more than the percentages specified in the individual monograph.