

Appendix XIV: Determination of Acid Value

Rancidity may occur in CMM samples upon storage especially when CMM contains high content of fatty acid or fatty oils. The decomposed components such as free fatty acids, peroxides, low molecular weight aldehydes and low molecular weight of ketones are produced. This would result in distinctive smell and affect the quality of the CMM samples. In view of this, acid value which is defined as the number of mg of potassium hydroxide required to neutralize the free acid in 1g of fat, fatty oil or other related substances is determined to assess the rancidity of the CMM samples. Sodium hydroxide may also be used.

Method and procedure

(1) Extraction of fatty oil

Pulverize 30-50 g of CMM sample, pass through No.2 sieve and mix well. Put the powdered sample in a cellulose extraction thimble. Place the cellulose extraction thimble in a soxhlet extractor. Add 100-150 mL of n-hexane to a 500-mL round-bottomed flask. Perform the soxhlet extraction using water bath for about 2 h. Cool down to room temperature. Filter through a No.3 sintered funnel. Transfer the filtrate to a 250-mL round-bottomed flask. Evaporate the solvent to dryness at reduced pressure in a rotary evaporator. Collect the fatty oil.

(2) Determination of acid value

(a) Reagents

Phenolphthalein indicator

Weigh 1 g of phenolphthalein and dissolve in 100 mL of ethanol.

Sodium hydroxide titrant

Weigh accurately 4.0 g of sodium hydroxide and place it in a 1000-mL volumetric flask. Make up to the mark with water.

Ethanol-ether solution

Prepare a mixture of ethanol and diethyl ether (1:1, v/v). Neutralize with sodium hydroxide titrant and add 1.0 mL of phenolphthalein indicator until pink colouration is observed. Freshly prepare the solution.

(b) Standardization of sodium hydroxide titrant

Weigh accurately 0.6 g of potassium hydrogen phthalate, previously dried to constant weight at 105°C, and place it in a 250-mL conical flask, then add 50 mL of water. Shake it well. Add 2 drops of phenolphthalein indicator. Titrate the solution with the sodium hydroxide titrant until pink colouration can be observed. Towards the end of titration, potassium hydrogen phthalate should be completely dissolved. Calculate the concentration of the sodium hydroxide titrant according to the following equation:

$$C_{\text{NaOH}} = \frac{W_{\text{C}_8\text{H}_5\text{KO}_4} \times P_{\text{C}_8\text{H}_5\text{KO}_4} \times 1000}{V_{\text{NaOH}} \times \text{Mw}_{\text{C}_8\text{H}_5\text{KO}_4}}$$

- where C_{NaOH} = Molarity of sodium hydroxide titrant (mol/L)
 V_{NaOH} = Volume of sodium hydroxide titrant used (mL)
 $\text{Mw}_{\text{C}_8\text{H}_5\text{KO}_4}$ = Molecular weight of potassium hydrogen phthalate (204.22 g)
 $W_{\text{C}_8\text{H}_5\text{KO}_4}$ = Weight of potassium hydrogen phthalate used (g)
 $P_{\text{C}_8\text{H}_5\text{KO}_4}$ = Purity of potassium hydrogen phthalate (%)

(c) Titration of test solution

Unless otherwise specified, weigh accurately a quantity of the fatty oil being examined as indicated in Table 1 and place it in a 250-mL conical flask, then add 50 mL of ethanol-ether solution. Shake it well. If necessary, reflux the mixture gently until the substance is completely dissolved. Titrate the solution with sodium hydroxide titrant until pink colouration can be observed which persists for 30 s. Measure the volume of sodium hydroxide titrant used and calculate the acid value according to the following equation:

$$\text{Acid value} = \frac{V_{\text{NaOH}} \times 5.61}{W}$$

- where V_{NaOH} = Volume of sodium hydroxide titrant used (mL)
 W = Weight of the fatty oil being examined (g)

When the acid value is less than 10, it is suggested that a 10-mL semi-micro burette may be used for the titration.

Table 1 Weight of fatty oil being examined

Presumed acid value	Weight of fatty oil being examined / g
0.5	10
1	5
10	4
50	2
100	1
200	0.5
300	0.4

Limits-The acid value in CMM samples should not be more than the value specified in the individual monograph.