12.3 Gross energy of milk

Sample preparation

1) Take 5 ml of milk sample in a bomb crucible.

2) Keep it for drying at 40ºC as in case of urine in the vacuum drying oven.

3) 59. After drying, scratch with a needle and burn the sample in the bomb calorimeter as given above.

Calculation

The gross energy of milk sample may be calculated using the following formula:

$$\text{GE (cal/ml)} = \frac{(\text{Bomb equivalent} \times T) \times A}{\text{Volume of milk (ml)}}$$

Correction for nitrogen and sulphur

1) After the sample is burnt, open the bomb, wash all interior surfaces of bomb with distilled water (Fig. 12.2).

2) Collect the washings in 250 ml beaker for estimation of H_2SO_4 and HNO_3 formed during combustion from sulphur and nitrogen.

3) Boil the washings collected in the beaker for about 5 minutes.

4) Cool and titrate against N/10 $Ba(OH)_2$ solution using phenolphthalein indicator.

5) Add 20 ml N/10 Na_2CO_3 solution and boil again.

6) Cool the contents, filter through Whatman No.1 filter paper and wash with hot distilled water (2-3 washings).

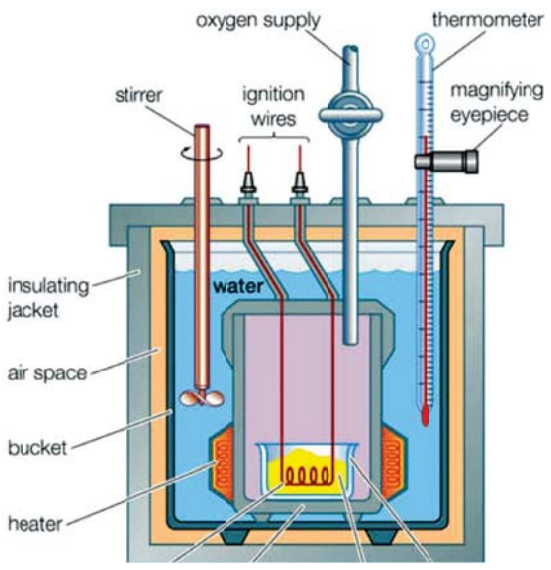

Fig. 12.2 Schematic diagram

7) Titrate the washings against N/10 HCl using methyl orange indicator.

8) Heat liberated by H_2SO_4 and HNO_3 can be calculated by using the following factors:

1 ml of N/10 $Ba(OH)_2$ solution = 3.60 cal
1ml of N/10 Na_2CO_3 solution = 1.43 cal

Calculations

a) Amount of N/10 Ba $(OH)_2$ solution used = A ml

b) Amount of N/10 Na_2CO_3 solution added = B ml

c) Amount of N/10 HCl used = C ml

Therefore, Nitric acid correction = 1.43 (B-C) cal

Sulphuric acid correction = 3.60 [A-(B-C)] cal

Ballistic oxygen bomb calorimeter

In this calorimeter, a know weight of samples is ignited electrically and burnt in an excess of oxygen in the bomb and the maximum temperature rise of the bomb is measured with the thermocouple and galvanometer system. By comparing this rise, with that obtained when a standard sample of known calorific value is burnt, the calorific value of the sample material can be determined. In this case, calibration constant is calculated by combusting benzoic acid in bomb calorimeter.

GE determination of feed and faeces

1) Take 1 g of ground, dried sample. Make the pellet of this using pelleting machine.

2) Weigh the empty steel crucible and then with sample.

3) Disconnect the thermocouple and remove the safety cover and bomb body.

4) Place the crucible on the support pillar in the base of the bomb.

5) Take one strand of 50 mm length of cotton, insert one end of the cotton between the coils of the firing wire and dip the other end into centre of the sample in the crucible.

6) Replace the safety cover and plug the thermo couple into hole in the top of the bomb body.

7) Fill the bomb with oxygen, keeping the pressure maximum up to 25 atmospheres.

8) By mean of “Galvo zero” known on the control box and the galvanometer mechanical zero arm, bring the light spot index of the galvanometer to zero, and leave for about 30 seconds to check that the temperature is stable.

9) Stand back from the bomb, press and release the firing button.

10) Note the maximum deflection of the galvanometer (which occurs about 40 seconds after firing).

For urine and milk

Sample preparation for urine and milk is same as in case of adiabatic bomb calorimeter described earlier. After the sample preparation, bombing of sample is done as described above from steps (2) to (10).

Standardizing the apparatus

1. **Correction for constant heat gain:** During every test a small, constant amount of heat is released in the bomb by firing cotton. To measure this, a test is carried out without any sample in the crucible.

2. **Calibration with standard sample:** This calibration is done to establish the relationship between the galvanometer deflection and the amount of heat released by the combustion of the sample. Thermo-chemical grade benzoic acid is recommended standard material but any other pure material such as salicylic acid or sugar of known

calorific value can be used. With benzoic acid, calorific value 6.32 kcal per g, about 0.7 g of benzoic acid is required.

Calculation of results

1)	Mass of benzoic acid	=	W g
2)	Calorific value of benzoic acid	=	6.32 kcal/g
3)	Heat released from benzoic acid	=	6.32 W kcal
4)	Galvanometer deflection without sample	=	O_1 divs
5)	Galvanometer deflection with benzoic acid	=	O_2 divs
6)	Deflection due to Benzoic acid and calibration constant	=	$\frac{6.32\ W}{O_2 - O_1} = y$

Gross energy of sample

1)	Mass of sample	=	Z gms
2)	Galvanometer deflection with sample	=	O_3 divs
3)	Galvanometer deflection without sample	=	O_1 divs
4)	Therefore, galvanometer deflection due to sample	=	O -O divs

5) Heat release from sample $= (O_3 - O_1)\ Y$ kcal

6) Calorific value of sample (kcal/g) $= \dfrac{(O_3 - O_1)\ Y}{Z}$

Reference: NUKAMP, H.J., 1965, “Some remarks about the determination of the heat of combustion and the carbon content of urine” fide EAAP, publication No.11, Editor, Blaxter, K.L., Academic Press, London, New York, pp.147-157.