# METHOD 19.0

Effective 1$^{st}$ January 2018

**CARBONATES**

**1.      Scope and Field of Application**
This method is for the determination of the carbonate content, expressed as calcium carbonate, of feeding stuffs.  This method should not be used in certain cases where, for example, iron carbonate is present.

**2.      Principle**
The sample is treated with hydrochloric acid and the carbon dioxide released is collected in a graduated tube, and its volume compared with that released by a known quantity of calcium carbonate under the same conditions.

**3.      Reagents**
**3.1**    Hydrochloric acid, density 1.1g/ml.
**3.2**    Calcium carbonate.
**3.3**    Sulphuric acid, approximately 0.05mol/litre, coloured with methyl red.

**4.      Apparatus**
Scheibler-Dietrich apparatus (fig. 1) or equivalent apparatus.

**5.      Procedure**
The quantity of sample taken for analysis depends on the supposed level of carbonate in the sample:
-   0.5g sample for contents from 50 to 100% of carbonates, expressed as calcium carbonate;
-   1g sample for contents from 40 to 50% of carbonates, expressed as calcium carbonate;
-   2 to 3g for contents less than 40%.

Place the portion of the sample in the special flask (D, in fig. 1) of the apparatus, fitted with a small tube of unbreakable material containing 10ml hydrochloric acid (3.1), and connect the flask to the apparatus. Turn the three-way tap (E) so that tube (A) connects with the outside.  Adjust the height of the mobile tube (B) containing coloured sulphuric acid (3.3) to bring the level of the liquid in the graduated tube (A) to the zero mark.  Turn the tap (E) in order to connect tube (A) with tube (C) and check that the level is at zero.

Tilt the flask (D) and slowly run the hydrochloric acid (3.1) over the portion of the sample.  Make the pressure equal by lowering the tube (B).  Shake the flask (D) until the release of carbon dioxide has ceased completely.  Restore pressure by bringing the liquid back to the same level in tubes (A) and (B).  Allow the volume of gas to become constant (several minutes) and then take the reading.  Carry out a control test using 0.5g of calcium carbonate (3.2) under the same conditions.

**6.       Expression of the Results**

**6.1**       Calculate the content of carbonates, expressed as calcium carbonate, using the formula

$$X = \frac{V \times 100}{V_1 \times 2m}$$

Where:

X          = % (w/w) of carbonates in the sample, expressed as calcium carbonate
V          = ml of $CO_2$ released by the portion of the sample
V1         = ml of $CO_2$ released by 0.5g of $CaCO_3$
m          = weight, in g, of the portion of the sample.

**7.       Observations**

**7.1**       When the portion of the sample weighs more than 2g, first place 15ml of distilled water in the flask (4.) and mix before beginning the text. Use the same volume of water for the control test.

**7.2**       If the apparatus used has a different volume from that of the Scheibler-Dietrich apparatus, the portions taken from the sample and from the control substance and the calculation of the results must be adapted accordingly.
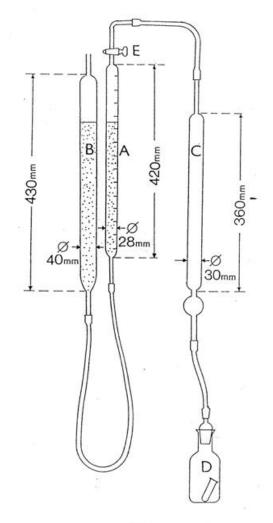
Fig. 1