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ANNEX

ANNEX

to the

Commission Implementing Regulation (EU) .../... of XXX

amending Regulation (EC) No 152/2009 as regards the determination of carbonates in feed

ANNEX

In Annex III to Commission Regulation (EC) No 152/2009, the following part is added after part ‘O. DETERMINATION OF CHLORINE FROM CHLORIDES’:

‘P. DETERMINATION OF CARBONATES

1. Purpose and Scope

This method makes it possible to determine the amount of carbonates, conventionally expressed as calcium carbonate, in feed with the exception of feed where iron carbnate is present.

2. Principle

The carbonates are decomposed in hydrochloric acid; the carbon dioxide released is collected in a graduated tube, and its volume compared with that released under the same conditions by a known quantity of calcium carbonate.

3. Reagents

3.1. Hydrochloric acid, density 1,10 g/ml.

3.2. Pure calcium carbonate.

3.3. Sulphuric acid, approximately 0,05 mol/litre, coloured with methyl red.

4. Apparatus

Scheibler-Dietrich apparatus (see diagram in the Appendix) or equivalent apparatus (calcimeter).

5. Procedure

Depending on the sample’s carbonate content, weigh a portion of the sample as shown below:

(a) 0,5 g for products containing from 50 % to 100 % of carbonates, expressed as calcium carbonate;
(b) 1 g for products containing from 40 % to 50 % of carbonates, expressed as calcium carbonate;
(c) 2 g to 3 g for other products.

Hydochloric acid (point 3.1, above) is added to the portion of the sample to decompose any carbonates present. The volume of carbon dioxide is measured by using a Scheibler-Dietrich apparatus or equivalent apparatus (calcimeter) and is compared with the volume of carbon dioxide produced by 0.5 g pure calcium carbonate (point 3.2, above).

All determinations shall be carried out under the same conditions in order to avoid making corrections for differences in temperature and pressure. The determination should be preferably carried out in a temperature-controlled room.

The procedure making use of the Scheibler-Dietrich apparatus is described in detail in the Appendix.

6. Calculation

The content of carbonates, expressed as pure calcium carbonate, is calculated by 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

V_1 = ml of CO_2 released by 0,5 g of CaCO3

m = weight, in grammes, of the portion of the sample.

7. Observations

7.1. If the Scheibler-Dietrich apparatus is used and the sample weighs more than 2 g, first place 15 ml of distilled water in the flask (item 4 in the diagram in the Appendix, below) and mix before beginning the test by adding hydrochlooric acid (point 3.1, above). Use the same volume of distilled 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 must be adapted accordingly.

Appendix

Detailed procedure making use of the Scheibler-Dietrich apparatus

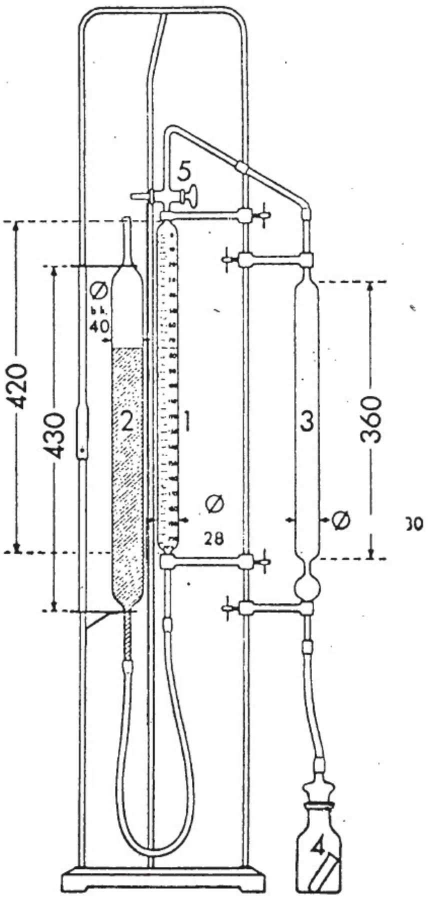
Place the portion of the sample in the special flask (item 4 in the diagram) of the apparatus, fitted with a small tube of unbreakable material containing 10 ml of hydrochloric acid (point 3.1, above), and connect the flask to the apparatus. Turn the three-way cock (item 5 in the diagram) so that the graduated tube (item 1 in the diagram) connects with the outside. Using the mobile tube (item 2 in the diagram), which is filled with coloured sulphuric acid (point 3.3, above) and is connected to the graduated tube (item 1 in the diagram), bring the level of the liquid up to the zero mark. Turn the three-way cock (item 5 in the diagram) in order to connect up the tubes (items 1 and 3 in the diagram) and check that the level is at zero.

Run the hydrochloric acid (point 3.1, above) slowly over the portion of the sample, tilting the special flask (item 4 in the diagram). Make the pressure equal by lowering the mobile tube (item 2 in the diagram). Shake the special flask (item 4 in the diagram) until the release of carbon dioxide has stopped completely.

Restore the pressure by bringing the liquid back to the same level in the tubes (items 1 and 2 in the diagram). After a few minutes, when the volume of gas has become constant, take the reading.

Carry out a control test in the same conditions on 0,5 g of calcium carbonate (point 3.2, above).

SCHEIBER-DIETRICH APPARATUS FOR THE DETERMINATION OF CO_2



(measured in mm)

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