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ANNEX

ANNEX

to the

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

**amending Regulation (EC) No 152/2009 laying down methods of sampling and analysis
for the official control of feed as regards the determination of carbonates**

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ANNEX

In Annex III to Commission Regulation (EC) 152/2009, the following part “P. DETERMINATION OF CARBONATES” 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 carbonate 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. Calcium carbonate.

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

4. Apparatus

Scheibler-Dietrich apparatus or equivalent apparatus.

5. Procedure

According to the sample's carbonate content, weigh a portion of the sample as shown below:

0,5 g for products containing from 50 to 100 % of carbonates, expressed as calcium carbonate;

1 g for products containing from 40 to 50% of carbonates, expressed as calcium carbonate;

2 to 3 g for other products.

Hydrochloric acid (3.1) is added to 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 (3.2.)

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.

As example, the procedure making use of the Scheibler-Dietrich apparatus is described in detail in the Appendix.

6. Calculation of results

The content of carbonates, expressed as 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 CO₂ released by the portion of the sample.

V₁ = ml of CO₂ 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 (4) in the diagram and mix before beginning the test by adding hydrochloric acid (3.1). 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.

Appendix

Detailed procedure making use of the Scheibler-Dietrich apparatus

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

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

Restore pressure by bringing the liquid back to the same level in tubes (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 (3.2).

SCHEIBLER-DIETRICH APPARATUS FOR THE DETERMINATION OF CO₂

