ANNEX

METHODS OF ANALYSIS OF THE COMPONENTS OF FEEDING-STUFFS

6. DETERMINATION OF ASH WHICH IS INSOLUBLE IN HYDROCHOLORIC ACID

1. Purpose and Scope

This method makes it possible to determine the level in feeding-stuffs of mineral substances which are insoluble in hydrochloric acid. Two methods can be used, depending on the nature of the sample.

1.1. *Method A:* applicable to straight organic feeding-stuffs and to most compound feeding-stuffs;

1.2. *Method B:* applicable to mineral compounds and mixtures and to compound feeding-stuffs whose content in substances insoluble in hydrochloric acid, as determined by Method A, is greater than 1 %.

2. Principle

2.1. *Method A:* the sample is ashed, the ash boiled in hydrochloric acid and the insoluble residue filtered and weighed.

2.2. *Method B:* the sample is treated with hydrochloric acid. The solution is filtered, the residue ashed and the ash thus obtained treated in accordance with Method A.

3. Reagents

3.1. Hydrochloric acid 3 N.

3.2. 20% solution (w/v) of trichloroacetic acid.

3.3. 1% solution (w/v) of trichloroacetic acid.

4. Apparatus

4.1. Hot plate.

4.2. Electric muffle-furnace with thermostat.

4.3. Crucibles for ashing made of platinum or an alloy of platinum and gold (10% Pt, 90% Au), either rectangular (60 × 40 × 25 mm) or circular (diameter 60 to 75 mm, height: 20 to 25 mm).

5. Procedure

5.1. Method A:

Ash the sample using the method described for the determination of crude ash. Ash obtained from that analysis may also be used.

Place the ash in a 250 to 400 ml beaker using 75 ml of hydrochloric acid 3 N (3.1). Bring slowly to the boil and boil gently for fifteen minutes. Filter the warm solution through an ash-free filter paper and wash the residue with warm water until the acid reaction is no longer visible. Dry the filter containing the residue and ash in a tared crucible at a temperature of not less than 550 ^{o}C and not more than 700 ^{o}C. Cool in a desiccator and weigh.

5.2. Method B

Weigh 5 g of the sample to the nearest mg and place in a 250 to 400 ml beaker. Add 25 ml of water and 25 ml of hydrochloric acid 3 N (3.1) successively, mix and wait for effervescence to cease. Add a further 50 ml of hydrochloric acid 3 N (3.1). Wait for any release of gas to cease then place the beaker in a boiling water bath and keep it there for thirty minutes or longer, if necessary, in order to hydrolyse thoroughly any starch which may be present.

Filter while warm through an ash-free filter and wash the filter in 50 ml of warm water (see observation 7). Place the filter containing the residue in a crucible for ashing, dry and ash at a temperature of not less than 550 ^{o}C and not more than 700 ^{o}C. Place the ash in a 250 to 400 ml beaker using 75 ml of hydrochloric acid 3 N (3.1); continue as described in the second subparagraph of 5.1.

6. Calculation of results

Calculate the weight of the residue by deducting the tare. Express the result as a percentage of the sample.

7. Observation

If filtration proves difficult recommence the analysis, replacing the 50 ml of hydrochloric acid 3 N (3.1) by 50 ml of 20% trichloroacetic acid (3.2) and washing the filter in a warm solution of 1% trichloroacetic acid (3.3).