1994 No. 309

AGRICULTURE

The Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1994

Made . . . . . 4th August 1994

Coming into operation . . . 16th September 1994

The Department of Agriculture, in exercise of the powers conferred on it by sections 66(1), 77(4), 78(6), 79(2), 84 and 86 of the Agriculture Act 1970(**a**), and of all other powers enabling it in that behalf, after consultation as required by section 84(1) of the said Act with such persons or organisations as appear to it to represent the interests concerned, hereby makes the following Regulations:

Title, commencement and interpretation

1.—(1) These Regulations may be cited as the Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1994 and shall come into operation on 16th September 1994.

(2) The Interpretation Act (Northern Ireland) 1954(**b**) shall apply to these Regulations as it applies to a Measure of the Northern Ireland Assembly.

Amendment of the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982

2. Schedule 2 to the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982(**c**) shall be further amended in accordance with paragraphs (*a*) to (*f*)—

(*a*) in paragraph 3 (**Methods of Analysis**)—

(i) item 16 shall be deleted;

(ii) for item 26a there shall be substituted the following item:

"26a. Aflatoxin B_1 — one-dimensional thin layer chromatographic method;"; and

(iii) for item 26b there shall be substituted the following item:

"26b. Aflatoxin B_1 — high performance liquid chromatographic method";

(*b*) for the provisions relating to method **4.** (**PROTEIN**), there shall be substituted the provisions set out in Schedule 1;

(**a**) 1970 c. 40; amended by the Agriculture Act 1970 Amendment Regulations 1982 (S.I. 1982/980)
(**b**) 1954 c. 33 (N.I.)
(**c**) S.R. 1982 No. 338 as amended by S.R. 1984 No. 26 and S.R. 1985 No. 194

(*c*) for the provisions relating to method **9. (FIBRE)**, there shall be substituted the provisions set out in Schedule 2;

(*d*) the provisions contained in method **16. (COPPER — DIETHYLDITHIOCARBAMATE SPECTROPHOTOMETRIC METHOD)**, shall be deleted;

(*e*) in method **26a. (AFLATOXIN B_1 — FOR CERTAIN STRAIGHT FEEDING STUFFS)**—

(i) for the heading and paragraph 1 there shall be substituted the following provisions:

"26a. AFLATOXIN B_1 — ONE-DIMENSIONAL THIN LAYER CHROMATOGRAPHIC METHOD

1. Scope and Field of Application

This method is for the determination of aflatoxin B_1 in raw materials and straight feeding stuffs. This method is not applicable to raw materials and straight feeding stuffs containing citrus pulp. The lower limit of determination is 0.01mg/kg (10 ppb). In the presence of interfering substances it is necessary to repeat the analysis using method 26b (high performance liquid chromatography)";

(ii) in paragraph 5.1 (*Defatting*) for the word "before" there shall be substituted the word "after";

(*f*) for the provisions relating to method **26b. (AFLATOXIN B_1 — FOR ALL OTHER FEEDING STUFFS)**, there shall be substituted the provisions set out in Schedule 3.

Sealed with the Official Seal of the Department of Agriculture for Northern Ireland on 4th August 1994.

(L.S.) *I. C. Henderson*
Assistant Secretary

SCHEDULE 1 Regulation 2(b)

"4. PROTEIN

1. Scope and Field of Application

This method is for the determination of the protein content of feeding stuffs on the basis of the nitrogen content, determined according to the Kjeldahl method.

2. Principle

The sample is digested by sulfuric acid in the presence of a catalyst. The acidic solution is made alkaline with sodium hydroxide solution. The ammonia is distilled and collected in a measured quantity of sulfuric acid, the excess of which is titrated with a standard solution of sodium hydroxide.

3. Reagents

3.1 Potassium sulfate.

3.2 Catalyst: copper (II) oxide CuO or copper (II) sulfate pentahydrate, $CuSO_4 - 5H_2O$.

3.3 Granulated zinc.

3.4 Sulfuric acid, $\rho_{20} = 1.34$ g/ml.

3.5 Sulfuric acid $c(\frac{1}{2}H_2SO_4) = 0.5$ mol/1.

3.6 Sulfuric acid $c(\frac{1}{2}H_2SO_4) = 0.1$ mol/1.

3.7 Methyl red indicator; dissolve 300 mg of methyl red in 100 ml of ethanol, $\sigma = 95–96\%$ (v/v).

3.8 Sodium hydroxide solution (Technical grade may be used) $\beta = 40$ g/100 ml (m/v: 40%).

3.9 Sodium hydroxide solution c = 0.25 mol/1.

3.10 Sodium hydroxide solution c = 0.1 mol/1.

3.11 Granulated pumice stone, washed in hydrochloric acid and ignited.

3.12 Acetanilide (m.p. = 114°C. N = 10.36%).

3.13 Sucrose (nitrogen free).

4. Apparatus

Apparatus suitable for performing digestion, distillation and titration according to the Kjeldahl procedure.

5. Procedure

5.1 *Digestion*

Weigh 1 g of the sample to the nearest 0.001 g and transfer the sample to the flask of the digestion apparatus. Add 15 g of potassium sulfate (3.1), an appropriate quantity of catalyst (3.2) (0.3 to 0.4 g of copper (II) oxide or 0.9 to 1.2 g of copper (II) sulfate pentahydrate), 25 ml of sulfuric acid (3.4) and a few granules of pumice stone (3.11) and mix. Heat the flask moderately at first, swirling from time to time if necessary until the mass has carbonized and the foam has disappeared; then heat more intensively until the liquid is boiling steadily. Heating is adequate if the boiling acid condenses on the wall of the flask. Prevent the sides from becoming overheated and organic particles from sticking to them. When the solution becomes clear and light green continue to boil for another two hours, then leave to cool.

5.2 *Distillation*

Add carefully enough water to ensure complete dissolution of the sulfates. Allow to cool and then add a few granules of zinc (3.3).

Place in the collecting flask of the distillation apparatus an exactly measured quantity of 25 ml of sulfuric acid (3.5) or (3.6) depending on the presumed nitrogen content. Add a few drops of methyl red indicator (3.7).

Connect the digestion flask to the condenser of the distillation apparatus and immerse the end of the condenser in the liquid contained in the collecting flask to a depth of at least 1 cm (see observation 8.3). Slowly pour 100 ml of sodium hydroxide solution (3.8) into the digestion flask without loss of ammonia (see observation 8.1).

Heat the flask until the ammonia has distilled over.

5.3 *Titration*

Titrate the excess sulfuric acid in the collecting flask with sodium hydroxide solution (3.9) or (3.10) depending on the concentration of the sulfuric acid used, until the end point is reached.

5.4 *Blank test*

To confirm that the reagents are free from nitrogen, carry out a blank test (digestion, distillation and titration) using 1 g of sucrose (3.13) in place of the sample.

6. Calculation of results

The content of protein is calculated according to the following formula:

$$\frac{(V_0 - V_1) \times c \times 0.014 \times 100 \times 6.25}{m}$$

where:

V_0 = Volume (ml) of NaOH (3.9 or 3.10) used in the blank test.

V_1 = Volumn (ml) of NaOH (3.9 or 3.10) used in the sample titration.

c = Concentration (mol/1) of sodium hydroxide (3.9 or 3.10).

m = Mass (g) of sample.

7. Verification of the method

7.1 *Repeatability*

The difference between the results of two parallel determinations carried out on the same sample must not exceed:

0.2% in absolute value, for protein contents of less than 20%;

1.0% relative to the higher value, for protein contents from 20% to 40%;

0.4% in absolute value, for protein contents of more than 40%.

7.2 *Accuracy*

Carry out the analysis (digestion, distillation and titration) on 1.5 to 2.0 g of acetanilide (3.12) in the presence of 1 g of sucrose (3.13); 1 g acetanilide consumes 14.80 ml of sulfuric acid (3.5). Recovery must be at least 99%.

8. Observations

8.1 Apparatus may be of the manual, semi-automatic or automatic type. If the apparatus requires transference between the digestion and distillation steps, this transfer must be carried out without loss. If the flask of the distillation apparatus is not fitted with a dropping funnel, add the sodium hydroxide immediately before connecting the flask to the condenser, pouring the liquid slowly down the side.

8.2 If the digest solidifies, recommence the determination using a larger amount of sulfuric acid (3.4) than that specified above.

8.3 For products with a low nitrogen content, the volume of sulfuric acid (3.6) to be placed in the collecting flask may be reduced, if necessary, to 10 or 15 ml and made up to 25 ml with water."