
STATUTORY RULES OF NORTHERN IRELAND

1999 No. 296

AGRICULTURE

**Feeding Stuffs (Sampling and Analysis)
Regulations (Northern Ireland) 1999**

Made - - - - *28th June 1999*

Coming into operation *2nd August 1999*

The Department of Agriculture, in exercise of the powers conferred by sections 66(1), 74A, 75(1), 76(1), 77(4), 78(6), 79(1), (2) and (9), 84 and 86 of the Agriculture Act 1970⁽¹⁾ and of every other power enabling it in that behalf, after consultation, in accordance with section 84(1) of that Act, with such persons or organisations as appear to it to represent the interests concerned, hereby makes the following Regulations:

Citation, commencement and interpretation

1.—(1) These Regulations may be cited as the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999, and shall come into operation on 2nd August 1999.

(2) In these Regulations “the Act” means the Agriculture Act 1970, and “the sampling Directive” means First Commission Directive [76/371/EEC](#) establishing the methods of sampling for the official control of feeding stuffs⁽²⁾.

(3) The Interpretation Act (Northern Ireland) 1954⁽³⁾ shall apply to these Regulations as it applies to a Measure of the Northern Ireland Assembly.

Prescribed amount for the purposes of the definition of sampled portion

2.—(1) The prescribed amount of material for the purposes of the definition of sampled portion in section 66(1) of the Act, so far as it relates to feeding stuffs, shall be determined in accordance with the provisions of this regulation.

(2) In relation to a solid feeding stuff in packages the prescribed amount shall be the quantity of material present or 5 tonnes, whichever is the less.

(3) In relation to a solid feeding stuff in bulk containers, the prescribed amount shall be—

(1) [1970 c. 40](#); section 74A was inserted by the European Communities Act [1972 \(c. 68\)](#), section 4(1) and Schedule 4, paragraph 6, and the Act was amended by the Agriculture Act 1970 Amendment Regulations 1982 (S.I. [1982/980](#))
(2) O.J. No. L102, 15.4.76, p. 1
(3) [1954 c. 33 \(N.I.\)](#)

- (a) the contents of the lowest number of containers which together hold not less than 5 tonnes; or
 - (b) if all the containers together hold less than 5 tonnes, or if all the feeding stuff is in one container, the quantity of material present; or
 - (c) if any container holds not less than 5 tonnes, the content of any such container.
- (4) In relation to a solid feeding stuff which is loose in heaps or bays, the prescribed amount shall be—
- (a) the contents of the lowest number of heaps or bays which together contain not less than 5 tonnes; or
 - (b) if all the heaps or bays together contain less than 5 tonnes, or if all the feeding stuff is in one heap or bay, the quantity of material present; or
 - (c) if any heap or bay contains not less than 5 tonnes, the content of any such heap or bay.
- (5) In relation to a liquid or semi-liquid feeding stuff in containers, the prescribed amount shall be—
- (a) the contents of the lowest number of containers which together hold not less than 5,000 litres; or
 - (b) if all the containers together hold less than 5,000 litres, or if all the feeding stuff is in one container, the quantity of material present; or
 - (c) if any container holds not less than 5,000 litres, the content of any such container.

Manner of taking, preparing, marking, sealing and fastening of samples

3. The manner in which samples of feeding stuffs are to be taken, prepared, marked, sealed and fastened, shall be as prescribed in Schedule 1 and paragraph 10 of Part II of that Schedule shall have effect for the purposes of the certificate referred to in regulation 6.

Methods of sending part of a sample

4. Any part of a sample of a feeding stuff required to be sent to any person in pursuance of subsection (1)(b) or (2) of section 77 of the Act shall be sent by registered post or by recorded delivery, or delivered or given by hand.

Application of methods of analysis

5.—(1) Subject to paragraph (2) for the purpose of determining whether a substance of a class or description—

- (a) listed in column 1 of Annex I to Part II to Schedule 2, or
- (b) to which the method of analysis specified in Annex II or the method specified in Annex III, to that Part relates,

is present or active in a sample of a feeding stuff to be analysed pursuant to the Act, or what quantity or proportion of such a substance is present or active as aforesaid, the provisions specified in Part I of that Schedule under the heading “GENERAL PROVISIONS” shall apply, and—

- (i) in relation to a substance of a class or description listed (whether by itself or by reference to its activity) in column 1 of Annex I to Part II of that Schedule, the relevant method of analysis set out in the European Community provision in force specified in the corresponding entry in columns 2 and 3 of that Annex shall be used, and
- (ii) in relation to a substance to which the method of analysis specified in Annex II, or the method specified in Annex III, to Part II of that Schedule relates, the method applicable to that

substance shall be used, and, where more than one method is set out in columns 2 and 3 of Annex I to Part II of that Schedule in relation to the same substance, the notes to that Annex shall have effect to specify which is the relevant method.

(2) After 31st October 1999, paragraph (1) shall cease to apply to the following substances listed in column 1 of Annex I to Part II of Schedule 2—

- (a) menadione (vitamin K3);
- (b) theobromine;
- (c) vitamin A; and
- (d) volatile mustard oil,

and shall cease to apply to starch insofar as it falls to be analysed by the pancreatic method as mentioned in the notes to that Annex.

(3) Where microscopic examination is carried out in order to—

- (a) determine whether any constituent of animal origin is present in a sample of a feeding stuff to be analysed pursuant to the Act; or
- (b) estimate the quantity of any such constituent present in any such sample,

then—

- (i) the general provisions specified in Part I of Schedule 2 shall apply, and
- (ii) the procedure set out in Commission Directive [98/88/EC](#) establishing guidelines for the microscopic identification and estimation of constituents of animal origin for the official control of feeding stuffs⁽⁴⁾ shall be used.

(4) Where a sample of a feeding stuff is to be analysed pursuant to the Act and neither paragraph (1) nor paragraph (3) applies—

- (a) if there is an applicable standard of the kind referred to in the first indent of Article 18.3 of Council Directive [95/53/EC](#) fixing the principles governing the organisation of official inspections in the field of animal nutrition⁽⁵⁾ (as amended), analysis shall be carried out in accordance with that standard,
- (b) if there is no such standard, it shall be carried out in accordance with any scientifically valid method the application of which does not contravene any general principle of the Treaty establishing the European Community.

Form of certificate of analysis

6. The certificate of analysis of any feeding stuff to be sent pursuant to section 77(4) of the Act shall be in the form set out in Part I of Schedule 3, and shall be completed in accordance with the notes set out in Part II of that Schedule.

Period within which analysis of the oil content of a feeding stuff must be carried out

7. Where a sample of a feeding stuff has been taken by an inspector in the prescribed manner and sent to an agricultural analyst for analysis, any analysis of the oil content of that feeding stuff shall be disregarded unless it is carried out before the expiry of the period of three weeks commencing with the date of sampling.

(4) O.J. No. L318, 27.11.98, p. 45

(5) O.J. No. L265, 8.11.95, p. 17 amended by Council Directive [1999/20/EC](#) (O.J. No. L80, 25.3.99, p. 20)

Modification of the Agriculture Act 1970 as respects metrication

8. For the purposes of its application to feeding stuffs, the Act shall continue to be modified as follows—

- (a) in the definition of sampled portion in section 66(1), the words “five tonnes or 5,000 litres” shall be substituted for the words “five tons or 1,000 gallons or the prescribed metric substitution”;
- (b) in section 68(2)(b), the words “to sales in quantities of not more than 25 kilograms” shall be substituted for the words “to sales of small quantities (that is to say, sales in quantities of not more than fifty-six pounds or the prescribed metric substitution)”; and
- (c) in section 76(5), the words “six kilograms” shall be substituted for the words “fourteen pounds or the prescribed metric substitution”.

Revocation

9. The Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982(6), the Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1984(7), the Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1985(8) and the Feeding Stuffs (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1994(9) are hereby revoked.

Sealed with the Official Seal of the Department of Agriculture for Northern Ireland on

L.S.

28th June 1999.

Liam McKibben
Assistant Secretary

(6) S.R. 1982 No. 338
(7) S.R. 1984 No. 26
(8) S.R. 1985 No. 194
(9) S.R. 1994 No. 309

SCHEDULE 1

Regulation 3

Manner of taking, preparing, marking, sealing and fastening of samples

PART I

Definitions

In this Schedule—

“sampled portion” means a quantity of a material constituting a unit and having characteristics presumed to be uniform;

“incremental sample” means a quantity taken from one point in the sampled portion;

“aggregate sample” means an aggregate of incremental samples taken from the same sampled portion;

“reduced sample” means a representative part of the aggregate sample obtained from the latter by a process of reduction;

“final sample” means a representative part of the reduced sample or, where no intermediate reduction is required, of the aggregate sample; and

“unit” has the same meaning as in the sampling Directive.

PART II

Instructions for the taking and preparation of Samples

1. In the case of feeding stuffs in packages or containers, except where section 68(2)(b) of the Act applies, only unopened packages or containers, which appear to the inspector proposing to take the sample to be the original packages or containers of the feeding stuff, shall be selected for the purpose of the sample.

2. The sample shall be taken and prepared as quickly as possible, having regard to the precautions necessary to ensure that it remains representative of the sampled portion. Instruments, surfaces and containers used in sampling shall be clean and dry.

3. No sample shall be drawn from any part of the sampled portion which appears to be damaged.

4. Where any appreciable portion of the feeding stuff appears to be mouldy, or is otherwise apparently unsuitable for feeding purposes, separate samples shall be drawn of the unsuitable portion and of the residue of the feeding stuff respectively. These shall be treated as separate sampled portions.

5.—(1) An inspector who intends to take a sample in accordance with the provisions of section 76(1)(b) of the Act, shall satisfy himself that the conditions in which the material concerned is stored are not such as might have caused undue deterioration thereof, and that it appears not to have been contaminated by any other material.

(2) The provisions of sub-paragraph (1) shall not apply as respects any feeding stuff purchased for the purpose of resale in the course of trade.

6. The sampling apparatus shall consist of materials which cannot contaminate the feeding stuff to be sampled.

7. Subject to paragraph 8, in the absence of good reason to the contrary, the sampling apparatus for solid feeding stuffs shall be taken from among the following—

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

- (a) a flat-bottomed shovel with vertical sides;
 - (b) a sampling spear with dimensions appropriate to the characteristics of the sampled portion in all respects, including dimensions of the container and particle size of the feeding stuff;
 - (c) mechanical apparatus which, if used for the purpose of sampling a feeding stuff being moved at the time the sample is taken, must be capable of taking samples right across the flow of the product;
 - (d) apparatus designed to divide the sample into approximately equal parts for taking incremental samples, and for the preparation of reduced and final samples.
8. A sampling spear shall not be used if the material is in a package or container containing not more than 50 kg and, prior to the taking of a sample, the manufacturer objects to such use on the ground that the material is unsuitable.
9. The sample shall be taken, prepared and packaged in accordance with the requirements specified, in the Annex to the Sampling Directive—
- (a) in paragraphs 5A and 5B under the heading “QUANTITATIVE REQUIREMENTS” (as set out in Section A of the Table to this Part); and
 - (b) in paragraphs 6.2 to 6.4 under the heading “INSTRUCTIONS FOR TAKING, PREPARING AND PACKAGING THE SAMPLES” (as set out in Section B of the Table to this Part).
10. Any sample taken in accordance with the preceding paragraphs shall be considered as representative of the sampled portion.

Table

Extracts from the Sampling Directive

Section A

Text referred to in Paragraph 9(a)

5.A.	In relation to the control of substances or products uniformly distributed throughout the feeding stuff	
5.A.1	<i>Sampled portion</i>	
	The size of the sampled portion must be such that each of its constituent parts can be sampled.	
5.A.2	<i>Incremental samples</i>	
5.A.2.1	Loose feeding stuffs:	Minimum number of incremental samples:
5.A.2.1.1.	Sampled portions not exceeding 2.5 metric tons	Seven
<p>(1) Where the number obtained is a fraction, it should be rounded up to the next whole number.</p> <p>(2) For packages or containers whose contents do not exceed 1 kg or one litre and for blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one original package or container, one block or lick.</p> <p>(3) The methods provided for in 5.A are for use in control of aflatoxins, rye ergot, castor-oil plant and crotalaria in complete and supplementary feeding stuffs.</p>		

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

5.A.2.1.2.	Sampled portions exceeding 2.5 metric tons	$\sqrt{}$ 20 times the number of metric tons making up the sampled portion ⁽¹⁾ , up to a maximum of 40 incremental samples
5.A.2.2.	Packaged feeding stuffs:	Minimum number of packages to be sampled ⁽²⁾
5.A.2.2.1	Packages of more than one kg:	
5.A.2.2.1.1.	Sampled portions of one to four packages	All packages
5.A.2.2.1.2.	Sampled portions of five to 16 packages	Four
5.A.2.2.1.3.	Sampled portions of more than 16 packages	$\sqrt{}$ Number of packages making up the sampled portion ⁽¹⁾ , up to a maximum of 20 packages
5.A.2.2.2.	Packages not exceeding 1 kg	Four
5.A.2.3.	Liquid or semi-liquid feeding stuffs:	Minimum number of containers to be sampled ⁽²⁾
5.A.2.3.1.	Containers of more than one litre:	
5.A.2.3.1.1.	Sampled portions of one to four containers	All containers
5.A.2.3.1.2.	Sampled portions of five to 16 containers	Four
5.A.2.3.1.3.	Sampled portions of more than 16 containers	$\sqrt{}$ Number of containers making up the sampled portion ⁽¹⁾ , up to a maximum of 20 containers
5.A.2.3.2.	Containers not exceeding one litre	Four
5.A.2.4.	Feed blocks and mineral licks	Minimum number of blocks or licks to be sampled ⁽²⁾
	One block or lick per sampled portion of 25 units, up to a maximum of four blocks or licks	
5.A.3.	<i>Aggregate sample</i>	

(1) Where the number obtained is a fraction, it should be rounded up to the next whole number.

(2) For packages or containers whose contents do not exceed 1 kg or one litre and for blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one original package or container, one block or lick.

(3) The methods provided for in 5.A are for use in control of aflatoxins, rye ergot, castor-oil plant and crotalaria in complete and supplementary feeding stuffs.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

A single aggregate sample per sampled portion is required. The total amount in the incremental samples making up the aggregate sample shall be not less than the following:

5.A.3.1.	Loose feeding stuffs	4 kg
5.A.3.2.	Packaged feeding stuffs:	
5.A.3.2.1.	Packages of more than 1 kg	4 kg
5.A.3.2.2.	Packages not exceeding 1 kg	Weight of the contents of four original packages
5.A.3.3.	Liquid or semi-liquid feeding stuffs:	
5.A.3.3.1.	Containers of more than one litre	Four litres
5.A.3.3.2.	Containers not exceeding one litre	Volume of the contents of four original containers
5.A.3.4.	Feed blocks or mineral licks:	
5.A.3.4.1.	Each weighing more than 1 kg	4 kg
5.A.3.4.2.	Each weighing not more than 1 kg	Weight of four original blocks or licks
5.A.4.	<i>Final samples</i>	

The aggregate sample gives the final samples on reduction when necessary. Analysis of at least one final sample is required. The amount in the final sample for analysis shall be not less than the following:

	Solid feeding stuffs	500 g
Liquid or semi-liquid feeding stuffs	500 ml	

5.B. In relation to the control of undesirable substances or products likely to be distributed non-uniformly throughout the feeding stuffs, such as aflatoxins, rye ergot, castor-oil plant and crotalaria in straight feeding stuffs⁽³⁾

5.B.1.	<i>Sampled portion:</i> see 5.A.1.	
5.B.2.	<i>Incremental samples</i>	
5.B.2.1.	Loose feeding stuffs: see 5.A.2.1.	
5.B.2.2.	Packaged feeding stuffs:	Minimum number of packages to be sampled

(1) Where the number obtained is a fraction, it should be rounded up to the next whole number.

(2) For packages or containers whose contents do not exceed 1 kg or one litre and for blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one original package or container, one block or lick.

(3) The methods provided for in 5.A are for use in control of aflatoxins, rye ergot, castor-oil plant and crotalaria in complete and supplementary feeding stuffs.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

- 5.B.2.2.1. Sampled portions consisting of one to four packages All packages
- 5.B.2.2.2. Sampled portions consisting of five to 16 packages Four
- 5.B.2.2.3. Sampled portions consisting of more than 16 packages $\sqrt{\text{Number of packages making up the sampled portion}^{(1)}}$, up to a maximum of 40 packages
- 5.B.3. *Aggregate samples*
- The number of aggregate samples will vary with the size of the sampled portion. The minimum number of aggregate samples per sampled portion is given below. The total weight of the incremental samples making up each aggregate sample shall be not less than 4kg.
- 5.B.3.1. Loose feeding stuffs
- | Size of the sampled portion in metric tons: | Minimum number of aggregate samples per sampled portion: |
|---|--|
| Up to 1 | 1 |
| More than 1 and up to 10 | 2 |
| More than 10 and up to 40 | 3 |
| More than 40 | 4 |
- 5.B.3.2. Packaged feeding stuffs size of the sampled portion in number of packages:
- | | |
|---------------|---|
| 1 to 16 | 1 |
| 17 to 200 | 2 |
| 201 to 800 | 3 |
| more than 800 | 4 |
- 5.B.4. *Final samples*
- Each aggregate sample gives the final samples on reduction. Analysis of at least one final sample per aggregate sample is required. The weight of the final sample for analysis may not be less than 500g.

-
- (1) Where the number obtained is a fraction, it should be rounded up to the next whole number.
- (2) For packages or containers whose contents do not exceed 1 kg or one litre and for blocks or licks weighing not more than 1 kg each, an incremental sample shall be the contents of one original package or container, one block or lick.
- (3) The methods provided for in 5.A are for use in control of aflatoxins, rye ergot, castor-oil plant and crotalaria in complete and supplementary feeding stuffs.
-

SECTION B

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

Text Referred to in Paragraph 9(b)

6.2.	Incremental samples
6.2.A.	<p><i>In relation to the control of substances or products uniformly distributed throughout the feeding stuff</i></p> <p>Incremental samples must be taken <i>at random throughout the whole sampled portion</i> and they must be of approximately equal sizes.</p>
6.2.A.1.	<p>Loose feeding stuffs</p> <p>A notional division shall be made of the sampled portion into a number of approximately equal parts. A number of parts corresponding to the number of incremental samples required in accordance with 5.A.2 shall be selected at random and at least one sample taken from each of these parts.</p> <p>Where appropriate, sampling may be carried out when the sampled portion is being moved (loading or unloading).</p>
6.2.A.2.	<p>Packaged feeding stuffs</p> <p>Having selected the required number of packages for sampling as indicated in 5.A.2, part of the contents of each package shall be removed using a spear or shovel. Where necessary, the samples shall be taken after emptying the packages separately.</p>
6.2.A.3.	<p>Homogeneous or homogenizable liquid or semi-liquid feeding stuffs</p> <p>Having selected the required number of containers for sampling as indicated in 5.A.2, the contents shall be homogenized if necessary and an amount taken from each container.</p> <p>The incremental samples may be taken when the contents are being discharged.</p>
6.2.A.4.	<p>Non-homogenizable, liquid or semi-liquid feeding stuffs</p> <p>Having selected the required number of containers for sampling as indicated in 5.A.2, samples shall be taken from different levels.</p>

-
- (1) For packaged feeding stuffs, a part of the contents of the packages to be sampled shall be removed, using a spear or shovel, after having, if necessary, emptied the packages separately.
 - (2) Any lumps shall be broken up (if necessary by separating them out and returning them to the sample) in each aggregate sample separately.
-

Samples may also be taken when the contents are being discharged but the first fractions should be discarded.

In either case the total volume taken must not be less than 10 litres.

6.2.A.5.

Feed blocks and mineral licks

Having selected the required number of blocks or licks for sampling as indicated in 5.A.2, a part of each block or lick shall be taken.

6.2.B.

In relation to the control of undesirable substances or products likely to be distributed non-uniformly throughout the feeding stuff, such as aflatoxins, rye ergot, castor-oil plant and crotalaria in straight feeding stuffs

A notional division shall be made of the sampled portion into a number or approximately equal parts, corresponding to the number of aggregate samples provided for in 5.B.3. If this number is greater than one, the total number of incremental samples provided for in 5.B.2 shall be distributed approximately equally over the different parts. Then samples of approximately equal sizes⁽¹⁾, and such that the total amount in the samples from each part is not less than the minimum 4kg quantity required for each aggregate sample, shall be taken. *Incremental samples taken from different parts shall not be aggregated.*

6.3.

Preparation of aggregate samples

6.3.A.

In relation to the control of substances of products distributed uniformly throughout the feeding stuff

The incremental samples shall be mixed to form a single aggregate sample.

6.3.B.

In relation to the control of undesirable substances or products likely to be distributed non-uniformly throughout the feeding stuff, such as aflatoxins, rye ergot, castor-oil plant and crotalaria in straight feeding stuffs

The incremental samples from each part of the sampled portion shall be mixed and the number of aggregate samples provided for in 5.B.3,

(1) For packaged feeding stuffs, a part of the contents of the packages to be sampled shall be removed, using a spear or shovel, after having, if necessary, emptied the packages separately.

(2) Any lumps shall be broken up (if necessary by separating them out and returning them to the sample) in each aggregate sample separately.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

made up *taking care to note the origin of each aggregate sample.*

6.4.

Preparation of final samples

The material in each aggregate sample shall be carefully mixed to obtain an homogenized sample⁽²⁾. If necessary the aggregate sample should first be reduced to at least 2kg or two litres (reduced sample) either by using a mechanical divider or by the quartering method.

At least three final samples shall then be prepared, of approximately the same amount and conforming to the quantitative requirements of 5.A.4 or 5.B.4. Each sample shall be put into an appropriate container. All necessary precautions shall be taken to avoid any change of composition of the sample, contamination or adulteration which might arise during transportation or storage.

-
- (1) For packaged feeding stuffs, a part of the contents of the packages to be sampled shall be removed, using a spear or shovel, after having, if necessary, emptied the packages separately.
- (2) Any lumps shall be broken up (if necessary by separating them out and returning them to the sample) in each aggregate sample separately.
-

PART III

Marking, Sealing and Fastening of the final Sample

1. Each container of a final sample shall be so secured and sealed by the person taking the sample that the container cannot be opened without breaking the seal; alternatively the container may be placed in a stout envelope or in a linen, cotton or plastic bag, and this further receptacle then secured and sealed in such a manner that the contents cannot be removed without breaking the seal or the receptacle.

2. A label shall be attached to the container or receptacle containing the final sample and sealed in such a manner that it cannot be removed without the seal being broken. The label shall be marked with the following particulars, which shall be visible without the seal being broken:

- (a) name of the inspector and the authority by which he was authorised to take the sample;
- (b) identification mark given by the inspector to the sample;
- (c) place of sampling;
- (d) date of sampling;
- (e) name of the material; and
- (f) identification code, batch reference number or consignment identification of the material sampled, where readily available.

3. The container or receptacle may also be secured and sealed by the holder of the material sampled or person acting on his behalf.

4. The label referred to above shall be signed or initialled by the person taking the sample or by or on behalf of the holder of the material sampled.

SCHEDULE 2

Regulation 5

Methods of Analysis

PART I

General Provisions

Introduction

- (a) (a) In general a single method analysis applies for the determination of the presence or quantity of a substance in feeding stuffs. Where two or more methods are prescribed the choice between them shall, except where otherwise indicated, be left to the agricultural analyst concerned; the method used must however be indicated in the certificate of analysis.
- (b) The result given in the analysis report shall be the average value obtained from at least two independent determinations, carried out on separate portions of the sample, and of satisfactory repeatability.
- (c) The result shall be expressed, in the manner laid down in the method of analysis, to an appropriate number of significant figures and shall be corrected, if necessary, to the moisture content of the final sample prior to preparation (see paragraph 3(d)).

Reagents and Apparatus

- (a) (a) Unless otherwise specified in the method of analysis concerned, all reagents must be analytically pure. The purity of the reagents, especially when determining trace elements, must be checked by a blank test. Depending upon the results obtained, further purification of the reagents may be required.
- (b) Where any operation involves preparation of solutions, dilution, rinsing or washing, as part of a method of analysis, water must be used unless the specification of the method indicates otherwise.
- (c) Water should, in the absence of good reason to the contrary, be demineralized or distilled. Where indicated in the method of analysis concerned it must be subjected to special purification procedures.
- (d) All instruments or apparatus used must be clean, especially when very small amounts of substances have to be determined.

Preparation of the sample for analysis

- (a) (a) Samples must be prepared in such a way that the amounts weighed out, as provided for in the methods of analysis, are homogeneous and representative of the final sample.
- (b) All the necessary operations must be performed in such a way as to avoid, as far as possible, any change in, or contamination of, the sample. Grinding, mixing and sieving should be carried out as quickly as possible with minimal exposure of the sample to the air and light. Overgrinding is to be avoided. Mills and grinders likely to heat the sample appreciably should not be used. Nevertheless, where some loss or gain of moisture is unavoidable, allowance should be made for such changes (see sub-paragraph (d)). Manual grinding is recommended for feeding stuffs which are particularly sensitive to heat. Care should also be taken to ensure that the apparatus itself is not a source of contamination by trace elements.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

- (c) If the final sample as received consists of unopened packages or containers then, immediately prior to the preparation of the sample for analysis, all the contents shall be thoroughly mixed together.
- (d) If the sample is appreciably moist, or if for any reason the preparation cannot be carried out without significant changes in the moisture content of the sample, determine the moisture content before and after preparation, using the method specified in columns 2 and 3 of Annex I to Part II of this Schedule, appearing opposite to the reference to “Moisture” in column 1 of that Annex.
- (e) When a microscopical examination for the presence of undesirable substances is required then, in the absence of good reason to the contrary—
 - (i) the sample should be crushed and ground only to such an extent that facilitates the examination, and
 - (ii) grinding to pass 1 mm should not be used where it could lead to difficulties in identifying the undesirable substances listed in Schedule 5 to the Feeding Stuffs Regulations (Northern Ireland) 1995⁽¹⁰⁾.

Procedure

- (f) Mix the sample thoroughly either mechanically or manually. Divide the sample into two equal portions (the quartering method should be used where applicable). Preliminary crushing and/or grinding may be necessary, if the sample is in a coarse condition, to facilitate this division. Keep one of the portions in a suitable container, i.e. non-corrodible, clean and dry and fitted with an air-tight stopper, and prepare the other portion or a representative part of it, of at least 100 g, as indicated below.
 - (i) *Feeding stuffs which can be ground as such*
 Unless otherwise specified in the method of analysis concerned, sieve the whole sample through a sieve having apertures of 1 mm square⁽¹¹⁾⁽¹²⁾, in accordance with recommendation ISO R565, after grinding, if necessary. Mix the sieved sample and collect it in a suitable container, i.e. non-corrodible, clean and dry and fitted with an air-tight stopper. Mix again, immediately before weighing out the amounts for analysis.
 - (ii) *Feeding stuffs which can be ground only after drying*
 Unless otherwise specified in the method of analysis concerned, dry the sample to reduce its moisture content to a level of 8-12%, in accordance with the preliminary drying procedure specified in point 4.3 of the method referred to in sub-paragraph (d) until grinding enables the sample to be passed wholly through a sieve having apertures of 1 mm square⁽¹¹⁾⁽¹²⁾. Then proceed as indicated in sub-paragraphs (f) (i).
 - (iii) *Liquid or semi-liquid feeding stuffs*
 Collect the sample in a suitable container, i.e. non-corrodible, clean and dry and fitted with an air-tight stopper. Mix thoroughly immediately before weighing out the amount for analysis.
 - (iv) *Other feeding stuff*

⁽¹⁰⁾ S.R. 1995 No. 451 as amended by S.R. 1996 No. 259, S.R. 1998 No. 124, S.R. 1998 No. 373 and S.R. 1999 No. 287

⁽¹¹⁾ Test sieves conforming to British Standard 410:1976 are suitable.

⁽¹²⁾ Where an analysis for copper has to be carried out, a stainless steel sieve should be used.

⁽¹¹⁾ Test sieves conforming to British Standard 410:1976 are suitable.

⁽¹²⁾ Where an analysis for copper has to be carried out, a stainless steel sieve should be used.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

A sample which cannot be prepared according to any of the above procedures should be treated by any other procedure which ensure that the amounts weighed out for analysis are homogenous and representative of the final sample.

Storage of samples

- (g) Samples must be stored at such a temperature as will cause no compositional changes. A sample intended for the analysis of vitamins, or substances which are particularly sensitive to light, should be placed in a brown glass container.

PART II

Methods of Analysis

The methods of analysis for the purposes of this Schedule are—

- (a) Community methods of analysis, as specified in Annex I to this Part of this Schedule;
 (b) the method for determining uric acid, as specified in Annex II to this Part of this Schedule;
 and
 (c) the method for determining isobutylidenediurea, as specified in Annex III to this Part of this Schedule.

ANNEX I

Community Methods of Analysis

Column 1 <i>Substance</i>	Column 2 <i>Community Provision</i>	Column 3 <i>Official Journal Reference</i>
Aflatoxin B ₁	Parts A and C of the Annex to Directive 76/372/EEC (Part A was replaced in part by paragraph I of the Annex to Directive 92/95/EEC. Part C was replaced entirely by the Annex to Directive 94/14/EC) ⁽¹⁾	O.J. No. L102, 15.4.76, p. 8. O.J. No. L327, 13.11.92, p. 54. O.J. No. L94, 13.4.94, p. 30.
Aflatoxin B ₁	Parts B and C of the Annex to Directive 76/372/EEC (Part B was replaced entirely by paragraph II of the Annex to Directive 92/95/EEC. Part C was replaced entirely by the Annex to Directive 94/14/EC) ⁽²⁾	O.J. No. L102, 15.4.76, p. 8. O.J. No. L327, 13.11.92, p. 54. O.J. No. L94, 13.4.94, p. 30.

(1) Where the one-dimensional thin layer chromatographic method is the appropriate one.
 (2) Where the high performance liquid chromatographic method is the appropriate one.
 (3) Where the pancreatic method is the appropriate one.
 (4) Where the polarimetric method is the appropriate one.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

Column 1 <i>Substance</i>	Column 2 <i>Community Provision</i>	Column 3 <i>Official Journal Reference</i>
Amino Acids	Part A of the Annex to Directive 98/64/EC	O.J. No. L257, 19.9.98, p. 14.
Ammonia and volatile nitrogenous bases	Part II of the Annex to Directive 71/393/EEC	O.J. No. L279, 20.12.71, p. 7 (O.J./S.E. 1971(III), p. 987).
Ash	Point 5 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Ash insoluble in hydrochloric acid	Point 6 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Calcium	Point 3 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Carbonates	Point 4 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Fibre	Point 3 of Annex 1 to Directive 73/46/EEC (as replaced entirely by the Annex to Directive 92/89/EEC)	O.J. No. L83, 30.3.73, p. 21. O.J. No. L344, 26.11.92, p. 35.
Free and total gossypol	Point 5 of Annex 1 to Directive 72/199/EEC	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-72 supplement, p. 74).
Hydrocyanic acid	Point 2 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Iron, copper, manganese and zinc	Point 3 of the Annex to Directive 78/633/EEC	O.J. No. L206, 29.7.78, p. 43.
Lactose	Point 9 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Magnesium	Point 2 of Annex 1 to Directive 73/46/EEC	O.J. No. L83, 30.3.73, p. 21.
Menadione (vitamin K ₃)	Point 5 of Annex II to Directive 74/203/EEC	O.J. No. L108, 22.4.74, p. 7.
Moisture	Part I of the Annex to Directive 71/393/EEC (as	O.J. No. L279, 20.12.71, p.7

(1) Where the one-dimensional thin layer chromatographic method is the appropriate one.

(2) Where the high performance liquid chromatographic method is the appropriate one.

(3) Where the pancreatic method is the appropriate one.

(4) Where the polarimetric method is the appropriate one.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

Column 1 <i>Substance</i>	Column 2 <i>Community Provision</i>	Column 3 <i>Official Journal Reference</i>
	amended by Article 1 of Directive 73/47/EEC)	(O.J./S.E. 1971(III), p. 987). O.J. No. L83, 30.3.73, p. 35.
Moisture in fats and oils	Point 1 of Annex 1 to Directive 73/46/EEC	O.J. No. L83, 30.3.73, p. 21.
Oils and fats	Part IV of the Annex to Directive 71/393/EEC (Part IV was replaced entirely by Annex I to Directive 84/4/EEC . That Annex was in turn replaced entirely by Part B of the Annex to Directive 98/64/EC) O.J. No. L257, 19.9.98, p. 14.	O.J. No. L279, 20.12.71, p. 7 (O.J./S.E. 1971(III), p. 987). O.J. No. L15, 18.1.84, p. 28.
Pepsin activity	Point 4 of Annex 1 to Directive 72/199/EEC	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-1972 supplement, p. 74).
Phosphorus	Part III of the Annex to Directive 71/393/EEC	O.J. No. L279, 20.12.71, p. 7 (O.J./S.E. 1971(III), p. 987).
Potassium	Point 10 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Protein	Point 2 of Annex 1 to Directive 72/199/EEC (as replaced entirely by the Annex to Directive 93/28/EEC)	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-1972 supplement, p. 74). O.J. No. L179, 22.7.93, p. 8.
Proteins soluble in pepsin and hydrochloric acid	Point 3 of Annex 1 to Directive 72/199/EEC	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-1972 supplement, p. 74).
Sodium	Point 11 of the Annex to Directive 71/250/EEC (as corrected by a corrigendum published in July 1975)	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480). Consolidated edition of corrigenda to the first series

- (1) Where the one-dimensional thin layer chromatographic method is the appropriate one.
- (2) Where the high performance liquid chromatographic method is the appropriate one.
- (3) Where the pancreatic method is the appropriate one.
- (4) Where the polarimetric method is the appropriate one.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

Column 1 <i>Substance</i>	Column 2 <i>Community Provision</i>	Column 3 <i>Official Journal Reference</i>
		of special editions of EC legislation (1952 to 1972).
Sugar	Point 12 of the Annex to Directive 71/250/EEC (as corrected by a corrigendum published in July 1975)	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480). Consolidated edition of corrigenda to the first series of special editions of EC legislation (1952 to 1972).
Starch	Annex 1 to Directive 74/203/EEC ⁽³⁾	O.J. No. L108, 22.4.74, p. 7.
Starch	Point 1 of Annex 1 to Directive 72/199/EEC (as corrected by a corrigendum published on 27 November 1980) ⁽⁴⁾	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-1972 supplement, p. 74).
		O.J. No. L320, 27.11.80, p. 43.
Theobromine	Point 13 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Urea	Point 14 of the Annex to Directive 71/250/EEC (as corrected by a corrigendum published in July 1975)	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480). Consolidated edition of corrigenda to the first series of special editions of EC legislation (1952 to 1972).
Urease activity	Point 16 of the Annex to Directive 71/250/EEC (as corrected by a corrigendum published in July 1975)	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480). Consolidated edition of corrigenda to the first series of special editions of EC legislation (1952 to 1972).
Vitamin A	Point 1 of Annex II to Directive 73/46/EEC	O.J. No. L83, 30.3.73, p. 21.
Volatile mustard oil	Point 8 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13

(1) Where the one-dimensional thin layer chromatographic method is the appropriate one.

(2) Where the high performance liquid chromatographic method is the appropriate one.

(3) Where the pancreatic method is the appropriate one.

(4) Where the polarimetric method is the appropriate one.

Column 1 <i>Substance</i>	Column 2 <i>Community Provision</i>	Column 3 <i>Official Journal Reference</i>
Water soluble chlorides	Point 7 of the Annex to Directive 71/250/EEC	O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
<p>(1) Where the one-dimensional thin layer chromatographic method is the appropriate one.</p> <p>(2) Where the high performance liquid chromatographic method is the appropriate one.</p> <p>(3) Where the pancreatic method is the appropriate one.</p> <p>(4) Where the polarimetric method is the appropriate one.</p>		

ANNEX II

Method for Determining Uric Acid

1 Scope and Field of Application

1. This method is for the determination of uric acid and its salts in dried poultry waste and in feeding stuffs containing dried poultry waste.

Principle

2. Uric acid is extracted with neutral ethanolic formaldehyde solution, precipitated as silver magnesium urate, redissolved in sodium thiosulphate solution and determined spectrophotometrically.

3 Reagents

3

3.1 Sodium hydroxide solution: dissolve 50 g sodium hydroxide in 50 ml water, mix well and store in a suitable plastic container.

3.2 Formaldehyde solution: the strength of the commercially available solution should be checked as follows: mix 3 ml formaldehyde solution with 50 ml 1N sodium hydroxide solution and 25 ml hydrogen peroxide solution (20 volumes). Heat on a steam bath until effervescence stops. Cool, and titrate with 1N hydrochloric acid using phenolphthalein indicator. Carry out a blank titration using 3 ml water in place of the formaldehyde.

1 ml of 1N sodium hydroxide \equiv 0.0300 g formaldehyde

strength of formaldehyde solution \equiv

$(BT) \times 0.0300 \times 100 \text{ g per } 100 \text{ ml}$

where:

B = blank titre; and

T = sample titre.

3.3 Neutral ethanolic formaldehyde solution: mix an appropriate volume of formaldehyde solution (3.2) containing 17.5 g of formaldehyde with 250 ml water and 500 ml ethanol. Adjust the pH of the solution to 7.0 with 0.1N sodium hydroxide solution. Dilute to 1,000 ml with water, mix and again adjust the pH to 7.0 if necessary.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

3.4 Succinate buffer solution: dissolve by heating, 29.5 g of succinic acid in 750 ml water and 20 ml sodium hydroxide solution (3.1). Cool, add an appropriate volume of formaldehyde solution (3.2) containing 17.5 g of formaldehyde, mix well and adjust the pH to 6.0 with sodium hydroxide solution (3.1). Dilute to 1,000 ml with water, mix and again adjust the pH to 6.0 if necessary.

3.5 Sodium thiosulphate solution: 25 g sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) per 1,000 ml.

3.6 Silver lactate solution: dissolve, by heating, 3 g silver lactate in 50 ml water and 1 ml lactic acid. Dilute to 100 ml with water, filter, and store in dark glassware. Do not expose to strong light.

3.7 Ammoniacal magnesium solution: dissolve 8.75 g magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) and 17.5 g ammonium chloride in 50 ml water. Add 30 ml ammonia solution ($d = 0.88 \text{ g/ml}$) mix well and dilute to 100 ml with water.

3.8 Benedict and Hitchcock reagent: mix 35 ml silver lactate solution (3.6) with 15 ml ammoniacal magnesium solution (3.7). Add 50 ml ammonia solution ($d = 0.88 \text{ g/ml}$). Mix well. Prepare immediately before use.

3.9 Standard uric acid solution: weigh to the nearest 0.1 mg, 250 mg of uric acid and transfer to a 150 ml round-bottomed flask fitted with a reflux condenser. Add 100 ml ethanolic formaldehyde solution (3.3) and boil under reflux on a steam bath for 30 minutes, shaking frequently. Cool, transfer the solution to a 250 ml graduated flask, wash the round-bottomed flask with ethanolic formaldehyde solution (3.3) and combine the washings with the uric acid solution. Dilute to the mark with ethanolic formaldehyde solution (3.3) and mix. 1 ml contains 1 mg of uric acid.

3.10 Light petroleum, boiling range 40-60 161 C.

4 Apparatus

4

4.1 Spectrophotometer, with 10 mm silica cells.

4.2 Percolation tubes, glass. Upper part: approximately 240 mm long, 18 mm internal diameter; lower part: approximately 120 mm long, 8 mm internal diameter.

5 Procedure

5

5.1 *Extraction of Uric Acid*

From dried poultry waste:

(5.1.1) Weigh to the nearest 0.001 g, about 0.4 g dried poultry waste and place in a 150 ml round-bottomed flask. Add 60 ml ethanolic formaldehyde solution (3.3), fit a reflux condenser onto the flask and heat on a steam bath for 1 hour. Cool and filter by suction through a sintered glass crucible (porosity 4) into a 100 ml graduated flask. Wash out the round-bottomed flask with 3 2 10 ml portions of ethanolic formaldehyde solution (3.3) passing each portion through the crucible into the graduated flask. Dilute to 100 ml with ethanolic formaldehyde solution and mix.

From feeding stuffs:

(5.1.2) Weigh to the nearest 0.001 g, between 4 g and 5 g of prepared sample. Transfer to a glass percolation tube (4.2) fitted with a small paper cup to retain the feed. Remove the fat from the feed by extraction with light petroleum (3.10). Transfer quantitatively the defatted sample to a 150 ml round-bottomed flask and remove the residual solvent with a slow current of air. Continue as in 5.1.1, second sentence “. . . Add 60 ml ethanolic formaldehyde solution (3.3) . . .”.

Determination

5.2 Transfer by pipette 20 ml of the sample extract prepared as in 5.1.1 or 5.1.2 to a 50 ml centrifuge tube. Add 10 ml of Benedict and Hitchcock reagent (3.8), mix well and allow to stand in the dark for 1 hour. Centrifuge at 2,000 rpm for 15 minutes, pour off the supernatant liquid and allow to drain for 10 minutes. Carefully wipe off any remaining liquid without disturbing the precipitate, and add 20-ml sodium thiosulphate solution (3.5) to each tube. Dissolve the precipitate by stirring with a thin glass rod. Transfer by pipette 5 ml of this solution into a 200 ml graduated flask containing 40 ml succinate buffer solution (3.4). Dilute to 200 ml with water and mix well. Measure the absorbance of the solution at 294 nm in 10 mm silica cells against a solution prepared by mixing 5 ml sodium thiosulphate solution (3.5) with 40 ml succinate buffer solution (3.4) and diluting to 200 ml with water. Determine the quantity of uric acid present by reference to the calibration curve (5.3).

Calibration Curve

5.3 Into a series of 50 ml centrifuge tubes, transfer by pipette 2, 4, 6, 8, 10 and 12 ml standard uric acid solution (3.9) (corresponding to 2, 4, 6, 8, 10 and 12 mg of uric acid) and make up to 20 ml with ethanolic formaldehyde solution (3.3). Add to each tube 10 ml Benedict and Hitchcock reagent (3.8), mix well and stand in the dark for 1 hour. Continue as in 5.2 from "... Centrifuge at 2,000 rpm. ...". Measure the absorbances of the solutions and plot the calibration curve using absorbances as the ordinates and the corresponding quantities of uric acid, in mg (as shown above) as the abscissae.

Expression of the Results

6. The uric acid nitrogen content per cent of the sample is given by the formula

$$A6 \times W$$

where:

A = mg uric acid (in the aliquot volume of the sample extract) as determined by photometric measurement; and

W = weight of sample in grams.

ANNEX III

Method for Determining Isobutylidenediurea

Scope and Field of Application

1. This method is for the determination of isobutylidenediurea in feeding stuffs.

Principle

2. The sample is hydrolysed, liberating isobutyraldehyde, the concentration of which is determined by gas chromatography.

Reagents

3.—(3.1) Toluene.

(3.2) Sodium sulphate, anhydrous.

(3.3) Buffer solution pH1: dissolve 27.2 g sodium acetate trihydrate in 300 ml 1M hydrochloric acid and add 700 ml water.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

(3.4) Buffer solution pH 0.65: dissolve 27.2 g sodium acetate trihydrate in 400 ml 1M hydrochloric acid and add 600 ml water.

(3.5) Isobutylidenediurea.

(3.6) Internal standard solution: dilute 5 ml isopropyl acetate to 100 ml with toluene (3.1).

Apparatus

4.—(4.1) 250 ml conical flasks with ground glass or PTFE stoppers.

(4.2) Stoppered centrifuge tubes.

(4.3) Gas chromatograph with flame ionisation detector.

(4.4) Column:

either (i) 1.5 m glass column (4 mm internal diameter) packed with 5% OV17 on Gas Chrom Q, 80-100 mesh,

or (ii) 1.5 m glass column (4 mm internal diameter) packed with 5% Carbowax 20M-TPA on Diatomite C-AAW, 80-100 mesh.

(4.5) Water bath: hotplate stirrer on which is placed a 2,000 ml beaker (or suitable vessel) containing water maintained at 40-50°C.

Procedure

Hydrolysis

Hydrolysis

5.—(5.1) Weigh to the nearest 0.001 g, between 3 and 7 g of the prepared sample containing about 0.2 g of isobutylidenediurea into a conical flask (4.1). Add 100 ml buffer solution (3.4) and 20.0 ml toluene (3.1) to the sample and place in the flask a magnetic bar. Stopper firmly to ensure that the flask remains tightly closed during the hydrolysis.

Place the flask in the water bath (4.5) and stir vigorously for 20 minutes. Remove the flask and immerse in an ice-water bath for 5 minutes. Add 15 g sodium sulphate (3.2) and 5.0 ml internal standard solution (3.6) to the contents of the flask. Stopper the flask again, shake, return to the water bath (4.5) and warm for 3 minutes with stirring. Cool in the ice-water bath for 5 minutes. Transfer slowly between 15 and 25 ml of the mixture to the centrifuge tube (4.2), stopper, and centrifuge for 5 minutes to separate the layers. (Repeat the transfer if insufficient toluene is decanted). Transfer a portion of the upper (toluene) layer to a test tube with a pasteur pipette.

Determination

(5.2) Inject between 0.5 and 1.0 ml of the toluene solution (5.1) into the gas chromatograph (4.3).

Suggested conditions:

Column	70°C	Nitrogen	40 ml per minute
Injection	150°C	Hydrogen	30 ml per minute
Detector	150°C	Air	370 ml per minute

Approximate retention times:

Isobutyraldehyde	1 min.
Internal standard	1.5 min.

Toluene

3 min.

Measure the peak heights of the isobutyraldehyde and internal standard. Calculate the peak height ratio, isobutyraldehyde/internal standard, and from this value determine the quantity of isobutylidenediurea present by reference to the calibration curve (5.3).

Calibration curve

(5.3) Weigh to the nearest mg, 100, 200 and 300 mg isobutylidenediurea (3.5) into three conical flasks (4.1). Add 100 ml buffer solution (3.3), 20.0 ml toluene (3.1) and a magnetic bar to each. Stopper the flasks firmly. Continue as in 5.1 from “... Place the flask in the water bath ...”. Inject the toluene solutions into the gas chromatograph (4.3), and measure the peak heights. Calculate the peak height ratios, isobutyraldehyde/internal standard, and plot the calibration curve using peak height ratios as the ordinates and the corresponding weights of isobutylidenediurea as the abscissae.

Expression of the Results

6. The per cent content of isobutylidenediurea in the sample is given by the formula:

$$AW \times 10$$

where:

A = weight of isobutylidenediurea (mg) read from the calibration curve; and

W = weight of sample in grams.

SCHEDULE 3

Regulation 6

Form of Certificate of Analysis

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

PART I

CERTIFICATE OF ANALYSIS OF FEEDING STUFF⁽¹⁾

I, the undersigned, an agricultural analyst in Northern Ireland, in pursuance of the provisions of Part IV of the Agriculture Act 1970, hereby certify that I received on the day of 19 , from⁽²⁾ one part of a sample of⁽³⁾ for analysis; which was duly sealed and fastened up and marked⁽⁴⁾ and was accompanied by a⁽⁵⁾ as follows:—⁽⁶⁾

and also by a signed statement that the sample was taken in the prescribed manner; and that the said part has been analysed by me, or under my direction, and I declare the results of analysis to be as follows:—⁽⁷⁾

⁽⁸⁾ Analysis for oil was completed on

⁽⁹⁾

and I am of the opinion that⁽¹⁰⁾

(A) specific method(s) is/are prescribed in the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999 for the analysis of (list substance(s)); and that/those method(s) was/were used in the analysis and/or

No specific method(s) is/are prescribed in the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999 for the analysis of (list substance(s)) and the method(s) used complied with regulation 5(4) of those Regulations⁽¹¹⁾.

Signature of analyst

Address

Date

PART II

NOTES FOR THE COMPLETION OF CERTIFICATE

(1) Statements made in certificates are to be confined to matters which are necessary to verify compliance with the Agriculture Act 1970.

(2) Insert the name of the inspector who submitted the sample for analysis; and also the mode of transit, for example "by hand", "by registered post", "by rail", as the case may be.

(3) Insert the name or description applied to the material.

(4) Insert the distinguishing mark on the sample and the date of sampling shown thereon.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

- (5) Insert either "statutory statement", "copy of statutory statement", "copy of particulars marked on the material" or "copy of particulars indicated by a mark applied to the material", or as the case may be.
- (6) Insert the particulars contained in the statutory statement, or particulars marked on or indicated by a mark applied to the material, or as the case may be.
- (7) Insert relevant results, including if appropriate—
- (a) the name and estimated percentage of any deleterious ingredient or undesirable substance found in the sample;
 - (b) the name or names of any additives found in the sample and whether or not they are permitted; and
 - (c) in the cases of those additives for which maxima are prescribed, whether the amounts found are in excess of the prescribed maxima.
- (8) In the case of a sample of any feeding stuff containing oil, insert the date of completion of the oil analysis.
- (9) In the case of analysis of substances for which no analytical method is prescribed in Schedule 2, indicate the method used. If analysis cannot be carried out because no suitable method exists, the certificate should be noted accordingly.
- (10) Enter information as follows—
- (a) whether the material was correctly named in accordance with the requirements of the Feeding Stuffs Regulations (Northern Ireland) 1995 and whether it accords with the meaning corresponding to that name; and if not, in what respect;
 - (b) if the composition of the material agrees with, or does not differ by more than the limits of variation from, the statement of particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, state that the particulars are correct within the limits of variation;
 - (c) if the composition of the material differs by more than the limits of variation from the statement of particulars contained in the statutory statement, or the particulars marked on or indicated by a mark associated with the material, or as the case may be, state the difference between the amount found and the amount stated, and that the difference is outside the limits of variation; and that the difference is to the prejudice of the purchaser, if such is believed to be the case;
 - (d) if the material is not suitable for the use as a feeding stuff having regard to section 72 of the Agriculture Act 1970, state in what respect.
- (11) Delete as applicable.

EXPLANATORY NOTE

(This note is not part of the Regulations.)

1. These Regulations consolidate with amendments the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1982 as amended and implement in Northern Ireland—

- (a) in full, the following European Community Directives—

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

First Commission Directive [71/250/EEC](#) (O.J. No. L155, 12.7.71, p. 13) (O.J./S.E. 1971(II), p. 480) establishing Community methods of analysis for the official control of feeding stuffs;

Second Commission Directive [71/393/EEC](#) (O.J. No. L279, 12.12.71, p. 7) (O.J./S.E. 1971 (III), p. 987) establishing Community methods of analysis for the official control of feeding stuffs;

Fourth Commission Directive [73/46/EEC](#) (O.J. No. L83, 30.3.73, p. 21) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [73/47/EEC](#) (O.J. No. L83, 30.3.73, p. 35) amending the second Commission Directive of 18 November 1971 establishing Community methods of analysis for the official control of feeding stuffs;

Seventh Commission Directive [76/372/EEC](#) (O.J. No. L102, 15.4.76, p. 8) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [92/89/EEC](#) (O.J. No. L344, 26.11.92, p. 35) amending Annex I to Fourth Directive [73/46/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [92/95/EEC](#) (O.J. No. L327, 13.11.92, p. 54) amending the Annex to the Seventh Directive [76/372/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [93/28/EEC](#) (O.J. No. L179, 22.7.93, p. 8) amending Annex I to the Third Directive [72/119/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [94/14/EC](#) (O.J. No. L94, 13.4.94, p. 30) amending Seventh Directive [76/372/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [98/88/EC](#) (O.J. No. L318, 27.11.98, p. 45) establishing guidelines for the microscopic identification and estimation of constituents of animal origin for the official control of feeding stuffs;

- (b) save insofar as they relate to the determination of zootechnical additives in feeding stuffs, the following European Community Directives—

Third Commission Directive [72/199/EEC](#) (O.J. No. L123, 29.5.72, p. 6) establishing Community methods of analysis for the official control of feeding stuffs;

Fifth Commission Directive [74/203/EEC](#) (O.J. No. L108, 22.4.74, p. 7) establishing Community methods of analysis for the official control of feeding stuffs;

First Commission Directive [76/371/EEC](#) (O.J. No. L102, 15.4.76, p. 1) establishing Community methods of sampling for the official control of feeding stuffs;

Eighth Commission Directive [78/633/EEC](#) (O.J. No. L206, 29.7.78, p. 43) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [81/680/EEC](#) (O.J. No. L246, 29.8.81, p. 32) amending Directives [71/250/EEC](#), [71/393/EEC](#), [72/199/EEC](#), [73/46/EEC](#), [74/203/EEC](#), [75/84/EEC](#), [76/372/EEC](#) and [78/633/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Commission Directive [84/4/EEC](#) (O.J. No. L15, 18.1.84, p. 28) amending Directives [71/393/EEC](#), [72/199/EEC](#) and [78/633/EEC](#) establishing Community methods of analysis for the official control of feeding stuffs;

Article 18.3 of Council Directive [95/53/EC](#) (O.J. No. L265, 8.11.95, p. 17) fixing the principles governing the organisation of official inspections in the field of animal nutrition;

Commission Directive [98/54/EC](#) (O.J. No. L208, 24.7.98, p. 49) amending Directives [71/250/EEC](#), [72/199/EEC](#), [73/46/EEC](#) and repealing Directive [75/84/EEC](#);

Commission Directive [98/64/EC](#) (O.J. No. L257, 19.9.98, p. 14) establishing Community methods of analysis for the determination of amino acids, crude oils and fats, and olaquinox in feeding stuffs and amending Directive [71/393/EEC](#);

Commission Directive [1999/27/EC](#) (O.J. No. L118, 6.5.1999, p. 36) establishing Community methods of analysis for the determination of amprolium, diclazuril and carbadox in feeding stuffs and amending Directives [71/250/EEC](#), [73/46/EEC](#) and repealing Directive [74/203/EEC](#).

2. The principal changes made by the Regulations are as follows—
 - (a) certain provisions in Directive [76/371/EEC](#) on sampling of feeding stuffs are now fully implemented (regulation 3 and Schedule 1);
 - (b) EC requirements as to sampling of feeding stuffs contained in Article 18.3 of Directive [95/53/EC](#) are implemented (regulation 3 and Schedule 1);
 - (c) parallel EC requirements as to analysis of feeding stuffs contained in that Article are implemented (regulation 5 and Schedule 2);
 - (d) in accordance with new EC requirements—
 - (i) previous EC methods of analysis relating to the detection of lupin alkaloids and the determination of thiamine, ascorbic acid and dehydroascorbic acid are excluded, as are (with effect from 1st November 1999) EC methods for starch (pancreatic method), volatile mustard oil, theobromine, vitamin A and vitamin K3 (menadione),
 - (ii) a new method of analysis for the determination of amino acids is added and a revised method for oils and fats substituted for the existing one, and
 - (iii) procedures on the detection and estimation of animal constituents in feeding stuffs are introduced,
(regulation 5 and Schedule 2);
 - (e) particular national methods of analysis relating to magnesium (gravimetric method) and calcium (atomic absorption method) are no longer covered (regulation 5 and Schedule 2).
3. The Regulations also revoke the previous Regulations (regulation 9) and include minor and drafting changes.