Statutory Rules 1999 No. 296
Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999

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 -

(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.

5.A.	**In relation to the control of substances or products uniformly distributed throughout the feeding stuff**	
5.A.1	*Sampled portion*	
	The size of the sampled portion must be such that each of its constituent parts can be sampled.	
5.A.2	*Incremental samples*	
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
5.A.2.1.2.	Sampled portions exceeding 2.5 metric tons	√ 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	√ 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	√ 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.	*Aggregate sample*	
	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.	*Final samples*	
	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.	*Sampled portion:* see 5.A.1.	
5.B.2.	*Incremental samples*	
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
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	√ 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:	Minimum number of aggregate samples per sampled portion:
	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, caster-oil plant and crotalaria in complete and supplementary feeding stuffs.

SECTION B

Text Referred to in Paragraph 9(b)

6.2.	Incremental samples

6.2.A.	*In relation to the control of substances or products uniformly distributed throughout the feeding stuff* Incremental samples must be taken *at random throughout the whole sampled portion* and they must be of approximately equal sizes.
6.2.A.1.	Loose feeding stuffs 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. Where appropriate, sampling may be carried out when the sampled portion is being moved (loading or unloading).
6.2.A.2.	Packaged feeding stuffs 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.
6.2.A.3.	Homogeneous or homogenizable liquid or semi-liquid feeding stuffs 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. The incremental samples may be taken when the contents are being discharged.
6.2.A.4.	Non-homogenizable, liquid or semi-liquid feeding stuffs Having selected the required number of containers for sampling as indicated in 5.A.2, samples shall be taken from different levels. 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[4], 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, 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[5]. 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.[4]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.[5]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

1. Introduction

(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.

Column 1 *Substance*	*Column 2* *Community Provision*	*Column 3* *Official Journal Reference*
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)[8]	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)[9]	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.
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).

		O.J. No. L83, 30.3.73, p. 21.
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. 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. O.J. No. L279, 20.12.71, p.7
Moisture	Part I of the Annex to Directive 71/393/EEC (as 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. O.J. No. L279, 20.12.71, p. 7
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./S.E. 1971(III), p. 987). O.J. No. L15, 18.1.84, p. 28.

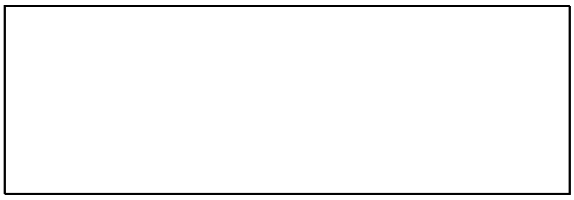
Pepsin activity	Point 4 of Annex 1 to Directive 72/199/EEC	O.J. No. L123, 29.5.72, p. 6 (O.J./S.E. 1966-1972 supplement, p. 74).
Phosphorus	Part III of the Annex to Directive 71/393/EEC	O.J. No. L279, 20.12.71, p. 7 (O.J./S.E. 1971(III), p. 987).
Potassium	Point 10 of the Annex to Directive 71/250/EEC	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 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[10]	O.J. No. L108, 22.4.74, p. 7. O.J. No. L123, 29.5.72, p. 6
Starch	Point 1 of Annex 1 to Directive 72/199/EEC (as corrected by a corrigendum published on 27 November 1980)[(11)]	(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. O.J. No. L155, 12.7.71, p. 13
Volatile mustard oil	Point 8 of the Annex to Directive 71/250/EEC	(O.J./S.E. 1971(II), p. 480).

		O.J. No. L155, 12.7.71, p. 13 (O.J./S.E. 1971(II), p. 480).
Water soluble chlorides	Point 7 of the Annex to Directive 71/250/EEC	[8]Where the one-dimensional thin layer chromatographic method is the appropriate one. [9]Where the high performance liquid chromatographic method is the appropriate one. [10]Where the pancreatic method is the appropriate one. [11]Where the polarimetric method is the appropriate one.

Notes:

[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)back
[2] O.J. No. L102, 15.4.76, p. 1back
[3] 1954 c. 33 (N.I.)back
[4] O.J. No. L318, 27.11.98, p. 45back
[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)back
[6] S.R. 1982 No. 338back
[7] S.R. 1984 No. 26back
[8] S.R. 1985 No. 194back
[9] S.R. 1994 No. 309back
[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. 287back



ANNEX II

Method for Determining Uric Acid

f Application

e determination of uric acid and its salts in dried poultry waste and in feeding stuffs containing dried poultry waste.

with neutral ethanolic formaldehyde solution, precipitated as silver magnesium urate, redissolved in sodium thiosulphate solution and
ly.

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

lution: the strength of the commercially available solution should be checked as follows: mix 3 ml formaldehyde solution with 50 ml 1N
ydrogen peroxide solution (20 volumes). Heat on a steam bath until effervescence stops. Cool, and titrate with 1N hydrochloric acid usi
blank titration using 3 ml water in place of the formaldehyde.

m hydroxide 0.0300 g formaldehyde
ldehyde solution
0 g

where:

nd

formaldehyde solution: mix an appropriate volume of formaldehyde solution (3.2) containing 17.5 g of formaldehyde with 250 ml wate
H 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 neces

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 appropria
n (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 w
o 6.0 if necessary.

ate solution: 25 g sodium thiosulphate ($Na_2S_2O_3.5H_2O$) per 1,000 ml.

tion: 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 gla
t.

gnesium solution: dissolve 8.75 g magnesium sulphate ($MgSO_4.7H_2O$) and 17.5 g ammonium chloride in 50 ml water. Add 30 ml ammo
nd dilute to 100 ml with water.

chcock reagent: mix 35 ml silver lactate solution (3.6) with 15 ml ammoniacal magnesium solution (3.7). Add 50 ml ammonia solution
iately before use.

d 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 condense
de solution (3.3) and boil under reflux on a steam bath for 30 minutes, shaking frequently. Cool, transfer the solution to a 250 ml gradu
k with ethanolic formaldehyde solution (3.3) and combine the washings with the uric acid solution. Dilute to the mark with ethanolic for
ontains 1 mg of uric acid.

, boiling range 40-60¡ C.

er, with 10 mm silica cells.

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

c Acid

ultry waste:

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),
ask 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
k with 3 2 10 ml portions of ethanolic formaldehyde solution (3.3) passing each portion through the crucible into the graduated flask. Di
de solution and mix.

tuffs:

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 fee
action with light petroleum (3.10). Transfer quantitatively the defatted sample to a 150 ml round-bottomed flask and remove the residua
Continue as in 5.1.1, second sentence " . . . Add 60 ml ethanolic formaldehyde solution (3.3) . . .".

0 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), r
l 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
e precipitate, and add 20-ml sodium thiosulphate solution (3.5) to each tube. Dissolve the precipitate by stirring with a thin glass rod. Tr
to a 200 ml graduated flask containing 40 ml succinate buffer solution (3.4). Dilute to 200 ml with water and mix well. Measure the abs
10 mm silica cells against a solution prepared by mixing 5 ml sodium thiosulphate solution (3.5) with 40 ml succinate buffer solution (
etermine the quantity of uric acid present by reference to the calibration curve (5.3).

e

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
h ethanolic formaldehyde solution (3.3). Add to each tube 10 ml Benedict and Hitchcock reagent (3.8), mix well and stand in the dark f
entrifuge at 2,000 rpm. ...". Measure the absorbances of the solutions and plot the calibration curve using absorbances as the ordinates an
d, in mg (as shown above) as the abscissae.

where:
(in the aliquot volume of the sample extract) as determined by photometric measurement; and
mple in grams.

ANNEX III
Method for Determining Isobutylidenediurea

f Application
e determination of isobutylidenediurea in feeding stuffs.

ysed, liberating isobutyraldehyde, the concentration of which is determined by gas chromatography.

anhydrous.

H1: dissolve 27.2 g sodium acetate trihydrate in 300 ml 1M hydrochloric acid and add 700 ml water.

H 0.65: dissolve 27.2 g sodium acetate trihydrate in 400 ml 1M hydrochloric acid and add 600 ml water.

ea.

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

asks with ground glass or PTFE stoppers.

uge tubes.

ph with flame ionisation detector.

lass column (4 mm internal diameter) packed with 5% OV17 on Gas Chrom Q, 80-100 mesh,
s column (4 mm internal diameter) packed with 5% Carbowax 20M-TPA on Diatomite C-AAW, 80-100 mesh.
ate stirrer on which is placed a 2,000 ml beaker (or suitable vessel) containing water maintained at 40-50¡C.

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
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 hydrol

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 soc
al 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 minut
ater 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 m
at the transfer if insufficient toluene is decanted). Transfer a portion of the upper (toluene) layer to a test tube with a pasteur pipette.

d 1.0 ml of the toluene solution (5.1) into the gas chromatograph (4.3).

ions:

Nitrogen	40 ml per mi
Hydrogen	30 ml per mi
Air	370 ml per m

ention times:

1 min.
1.5 min.
3 min.

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

e

mg, 100, 200 and 300 mg isobutylidenediurea (3.5) into three conical flasks (4.1). Add 100 ml buffer
l toluene (3.1) and a magnetic bar to each. Stopper the flasks firmly. Continue as in 5.1 from ".... Place
bath ....". Inject the toluene solutions into the gas chromatograph (4.3), and measure the peak heights.
eight ratios, isobutyraldehyde/internal standard, and plot the calibration curve using peak height ratios as
corresponding weights of isobutylidenediurea as the abscissae.

Results

of isobutylidenediurea in the sample is given by the formula:

where:

obutyldenediurea (mg) read from the calibration curve; and

mple in grams.

SCHEDULE 3

Form of Certificate of Analysis

PART I

Certificate of Analysis of Feeding Stuff[12]

n agricultural analyst in Northern Ireland, in pursuance of the provisions of Part IV of the Agriculture

tify that I received on the day of 19 , from[13] one part of a sample of[14] for analysis; which was duly

p and marked[15] and was accompanied by a[16] as follows: - [17]

statement that the sample was taken in the prescribed manner; and that the said part has been analysed

direction, and I declare the results of analysis to be as follows: - [18]

s completed on

on that[21]

s) is/are prescribed in the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999

st substance(s)) and that/those method(s) was/were used in the analysis and/or

s) is/are prescribed in the Feeding Stuffs (Sampling and Analysis) Regulations (Northern Ireland) 1999

st substance(s)) and the method(s) used complied with regulation 5(4) of those Regulations[(22)] .

PART II

Notes for the Completion of Certificate

de in certificates are to be confined to matters which are necessary to verify compliance with the

.

of the inspector who submitted the sample for analysis; and also the mode of transit, for example "by post", "by rail", as the case may be.

or description applied to the material.

guishing mark on the sample and the date of sampling shown thereon.

atutory statement", "copy of statutory statement", "copy of particulars marked on the material" or "copy

ed by a mark applied to the material", or as the case may be.

culars contained in the statutory statement, or particulars marked on or indicated by a mark applied to

e case may be.

results, including if appropriate -

estimated percentage of any deleterious ingredient or undesirable substance found in the sample;