

SCHEDULE 2

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.

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- (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(1).

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(2)(3), 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(2)(3). 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*

(1) 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

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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.