

INTERNATIONAL STANDARD

ISO
9167-2

First edition
1994-10-01

Rapeseed — Determination of glucosinolates content —

Part 2:

Method using X-ray fluorescence
spectrometry

Graines de colza — Dosage des glucosinolates —

Partie 2: Méthode par spectrométrie de fluorescence aux rayons X



Reference number
ISO 9167-2:1994(E)
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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9167-2 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 2, *Oleaginous seeds and fruits*.

ISO 9167 consists of the following parts, under the general title *Rapeseed — Determination of glucosinolates content*:

- *Part 1: Method using high-performance liquid chromatography*
- *Part 2: Method using X-ray fluorescence spectrometry*

Annexes A and B of this part of ISO 9167 are for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Rapeseed — Determination of glucosinolates content —

Part 2: Method using X-ray fluorescence spectrometry

1 Scope

This part of ISO 9167 specifies a rapid method for the determination of the total glucosinolate content of rapeseed using X-ray fluorescence spectrometry (XRF).

It is applicable to seeds with a normal protein content from 19 % to 23 %. For seeds with a protein content outside this range, account should be taken of the total protein content in the calculation of the total glucosinolate content (see reference [4]).

NOTES

1 ISO 9167-1 specifies a method using high-performance liquid chromatography (HPLC) which enables the content of different glucosinolates to be determined individually.

2 When applied to immature seed or to seed that has been badly stored, for example under damp and warm conditions, the results obtained by the XRF method and by the HPLC method (ISO 9167-1) may not agree as closely as when applied to normal rapeseed. The HPLC method should be taken as the reference method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9167. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9167 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO

maintain registers of currently valid International Standards.

ISO 664:1990, *Oilseeds — Reduction of laboratory sample to test sample*.

ISO 665:1977, *Oilseeds — Determination of moisture and volatile matter content*.

3 Definition

For the purposes of this part of ISO 9167, the following definition applies.

3.1 total glucosinolate content: The total sulfur content minus those amounts that are bound in proteins or single glucosinolates that cannot be determined by direct reference methods, divided by the average stoichiometric number of sulfur atoms occurring in the glucosinolate fraction typical of *Brassica* species.

4 Principle

Determination of total sulfur content in ground and compressed rapeseed by X-ray fluorescence spectroscopy. Calculation of the glucosinolate content by comparison with values of reference samples with a certified sulfur content.

5 Materials

5.1 Whole rapeseed reference materials, three samples, each with a certified sulfur content.

5.2 Whole rapeseed secondary reference materials, three samples, each with a sulfur content which is traceable to certified whole rapeseed reference materials (5.1).

5.3 Synthetic calibration samples, three samples, covering the range of sulfur content of interest.

NOTE 3 These samples may be those provided by the manufacturer of the X-ray spectrometer or prepared from powdered material.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Spoon, allowing the taking of about 20 g (about 30 ml) of seed.

6.2 Blender, coffee-mill type of 100 cm³ volume, 8 cm diameter and 180 W power.

6.3 Spatulas.

6.4 Aluminium cups or liquid cuvettes, with windows of polycarbonate film.

6.5 Hydraulic press, or other device (**hand-press**) allowing repeatable application of a defined pressure.

6.6 X-ray spectrometer, of high dispersion (better than 50 eV) with vacuum equipment or helium atmosphere.

6.7 Ventilated oven, capable of operating at 85 °C ± 1 °C, or **microwave oven** of 650 W power, for drying seeds.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this part of ISO 9167. A recommended sampling method is given in ISO 542[1].

8 Preparation of samples

8.1 Preparation of test samples

8.1.1 Reduce the laboratory sample in accordance with the method given in ISO 664.

8.1.2 If the moisture content of the seeds exceeds 10 %, reduce this to between 7 % and 9 % by drying about 40 g (60 ml) of the seeds, spread out in a thin layer, either in the microwave oven (6.7) for 2 min or in the ventilated oven for 75 min at 85 °C.

Allow the seeds to cool to room temperature before proceeding.

8.1.3 Carry out a parallel determination of the moisture content in accordance with the method given in ISO 665.

8.2 Preparation of reference samples

8.2.1 Treat the rapeseed reference materials (5.1) as described in 8.1.2 and 8.1.3. Three examples of each reference sample are required.

8.2.2 Use these samples to calibrate the apparatus when it is first put into service and at each further calibration (see 9.3.2).

8.2.3 Use a new series of reference samples of rapeseed (5.1) for each new calibration procedure.

9 Procedure

9.1 Test portion

9.1.1 Take, by means of the spoon (6.1), about 20 g (30 ml) of dried rapeseed, and grind them for 30 s in the grinder (6.2). Scrape off any meal adhering to the walls of the blender by means of the spatula (6.3) and grind the sample again for no more than 1 s, to ensure homogenization.

9.1.2 Fill the aluminium cup or liquid cuvette (6.4) with homogenized meal to the upper edge, or up to the mark specified by the manufacturer. Handle cups or cuvettes gently after filling to avoid separation of particles of different sizes.

NOTE 4 The quantity of meal put into the aluminium cups or liquid cuvettes depends only on the type of cup or cuvette used and does not influence the results of the determination. However, for each laboratory and each given apparatus, a constant quantity should be used.

9.2 Compression of the meal

In all cases, including the reference samples (5.1), the pressure shall be kept constant to within ± 10 %.