

ISO 9167:2019 (E)

Rapeseed and rapeseed meals — Determination of glucosinolates content — Method using high-performance liquid chromatography

Contents

	Foreword
	Introduction
1	Scope
2	Normative references
3	Terms and definitions
4	Principle
5	Reagents
6	Apparatus
7	Sampling
8	Preparation of the test sample
9	Procedure
9.1	Test portion
9.2	Extraction of glucosinolates
9.3	Blank test
9.4	Preparation of ion-exchange columns
9.5	Purification and desulfatation
9.6	Chromatography with gradient elution
9.6.1	General
9.6.2	Adjustment of the apparatus
9.6.2.1	General
9.6.2.2	Analysis
9.6.2.3	Examination of chromatograms
9.6.2.3.1	Identification of the peaks
9.6.2.3.2	Quantification
10	Expression of results
10.1	Calculation of the content of each glucosinolate
10.2	Relative proportionality factors
10.3	Calculation of the total glucosinolate content
11	Precision
11.1	Interlaboratory test
11.2	Repeatability
11.3	Reproducibility
12	Test report
Annex A	(informative) Results of interlaboratory trials — Gradient elution HPLC method
Annex B	(normative) Checking of the titre of the prepared internal standard solution
B.1	Determination of purity
B.2	Determination of the titre
B.2.1	Principle

- B.2.2 Glucose release method
- B.2.3 Calibration by a reference material
- B.3 Relative proportionality factors

Annex C (normative) Preparation and test of purified sulfatase solution and checking of the desulphation step on ion-exchange columns

- C.1 General
- C.2 Principle
 - C.2.1 Purification
 - C.2.2 Activity measurement
 - C.2.3 Activity checking on ion exchange columns
- C.3 Reagents and apparatus
- C.4 Procedures
 - C.4.1 Method A purification
 - C.4.2 Method B purification
 - C.4.3 Activity measurement of the purified sulfatase
 - C.4.4 Preparation of “ready to use” solutions
 - C.4.5 Checking of the sulfatase activity on ion-exchange columns

Annex D (informative) HPLC system and column performance criteria qualification

- D.1 General
- D.2 Procedure
 - D.2.1 Sinigrin and glucotraopaeolin peak shapes
 - D.2.2 Resolution of internal standards
 - D.2.3 Critical resolution
- D.3 System suitability
 - D.3.1 General
 - D.3.2 System artefacts
 - D.3.3 Carryover — System precision

Annex E (informative) Elution in the isocratic mode

- E.1 Internal standard
- E.2 Mobiles phases for isocratic elution
- E.3 Column for isocratic elution
- E.4 Chromatography with isocratic elution (simplified method)
 - E.4.1 Adjustment of the apparatus and the eluent
 - E.4.2 Analysis
 - E.4.3 Examination of chromatograms
 - E.4.3.1 Identification of the peaks
 - E.4.3.2 Quantification
 - E.4.3.2 Quantification
- E.5 Expression of results
 - E.5.1 Calculation of the content of each glucosinolate
 - E.5.2 Relative proportionality factors
- E.6 Calculation of the total glucosinolate content
- E.7 Precision
 - E.7.1 Interlaboratory test
 - E.7.2 Repeatability
 - E.7.3 Reproducibility
- E.8 Test report
- E.9 Results of the interlaboratory trial for the HPLC isocratic elution method

Page count: 28