

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 glucotrapaeolin 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 Mobile 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.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