DIN EN 16274:2021-11 (E)

Method for analysis of allergens - Quantification of an extended list of 57 suspected allergens in ready to inject fragrance materials by gas chromatography mass spectrometry

Contents

Page

European foreword 4

Introduction 5

1 Scope 6

2 Normative references 6

3 Terms and definitions 6

4 Principle 6

5 Reagents 7
5.1 Solvents 7
5.1.1 Methyl pivalate 7
5.1.2 Tert-Butyl Methyl Ether (MTBE) 7
5.2 Reference samples (suspected allergens) 7
5.2.1 General 7

6 Apparatus 11
6.1 Gas chromatograph equipped with flame ionization detector (GC-FID) 11
6.2 Gas chromatograph coupled to a mass spectrometer (GC-MS) 11
6.2.1 General 11
6.2.2 GC-MS system 11
6.3 Capillary columns for GC 11
6.4 Analytical balance 12

7 Stock and sample solutions - preparation and storage 12
7.1 General information 12
7.1.1 Choice of the solvent 12
7.1.2 Miscellaneous 13
7.2 Preparation of stock solutions from reference samples 13
7.3 Preparation of internal standard solutions and calibration solutions 13
7.3.1 General 13
7.3.2 Stock Solutions of Internal Standards (1g/kg equivalent) 13
7.3.3 Calibration Solutions Concentrations 13
7.3.4 Preparation of Calibration solutions 14

8 Mass spectrometer acquisition conditions 16
8.1 Establishment of retention times and SIM chromatograph windows 16
8.2 SIM Window - Set up and criteria 17
8.3 GC-MS scan mode verification 17

9 Sample analysis 17
9.1 General 17
9.2 Sample preparation for analysis 17
9.3 Sequence 18
9.3.1 Blank 18
9.3.2 Calibration solutions 18

9.3.3 Check solution 18
9.3.4 Samples 19

10 Data validation and treatment 19
10.1 General 19
10.2 Examination of Q values 19
10.3 Relative intensity of characteristic ions in SIM 20
10.4 Relative ion intensity using scan data 20
10.5 Data verification scheme and reporting of final concentration 21
10.6 GC-MS scan mode verification 21
10.7 Mass spectrum evaluation in SCAN mode 21

11 Test report 21

Annex A (informative) Determination of calibrant and reference sample purity 22

Annex B (informative) GC capillary column parameters 29

Annex C (informative) SIM Ions for SIM or SIM-SCAN Use 30

Annex D (informative) Example of SIM windows 37

Annex E (informative) Example of chromatograms 46

Annex F (normative) Decisional tree for quantification of suspected allergens 48

Annex G (informative) Preparation of stock solutions from reference samples 49

Annex H (informative) Calibration methods and approach 54

Annex I (informative) Quantification of allergens 56

Annex J (informative) Suspected allergens selected as analytes targets - Rationale 59

Annex K (informative) Other general information 69

Bibliography 70