DIN EN 14791:2006-04 (E)

Stationary source emissions - Determination of mass concentration of sulphur dioxide - Reference method

Contents

Page

Foreword 5

1 Scope 6

2 Normative references 6

3 Terms, definitions, symbols and abbreviations 6
3.1 Terms and definitions 6
3.2 Symbols 11
3.3 Abbreviations 12

4 Principle 13
4.1 General 13
4.2 Measuring principle 13

5 Reagents 13
5.1 General 13
5.2 Hydrogen peroxide 13
5.3 Water 13
5.4 Absorption solution, H2O2 13
5.5 Reagents for chromatographic analysis 14
5.5.1 Eluent solution 14
5.5.2 Standard sulphate stock solution, 10,4x10-3 mol/l -2 4SO 14
5.5.3 Regeneration solution for suppressor 14
5.6 Reagent for Thorin analysis 14
5.6.1 2-propanol [CH3CH(OH)CH3] 14
5.6.2 Barium perchlorate, standard volumetric solution, [Ba(ClO4)2] = 0,005 mol/l 14
5.6.3 Potassium hydroxide, standard volumetric solution, [KOH] = 0,1 mol/l 15
5.6.4 Perchloric acid, standard volumetric solution, [HClO4] = 0,1 mol/l 15
5.6.5 Thorin, {4-[2-arsonophenyl)-azo]-3-hydroxy-2,7 naphthalene-disulfonic acid disodium salt} 2 g/l solution 15

6 Sampling equipment 15
6.1 General 15
6.2 Sampling probe 15
6.3 Filter housing 16
6.4 Particle filter 16
6.5 Temperature controller 16
6.6 Absorbers 16
6.6.1 General 16
6.6.2 Test of absorption efficiency 17
6.7 Sampling pump 17
6.8 Gas volume meter 17

7 Sampling procedure 18
7.1 General requirements 18
7.2 Preparation and installation of equipment 18
7.2.1 Sampling location 18
7.2.2 Sampling point(s) 18
7.2.3 Assembling the equipment 19
7.2.4 Heating of the sampling line 19

7.2.5 Leak test ... 19
7.3 Performing sampling ... 19
7.3.1 Introduction of the probe in the duct ... 19
7.3.2 Sampling ... 19
7.3.3 Rinsing of the sampling system and preparation of the samples ... 20
7.4 Measuring series ... 20
7.5 Field blank ... 20
7.6 Absorption efficiency ... 21

8 Analysis equipment ... 21
8.1 Ion chromatograph ... 21
8.2 Thorin method ... 22
8.2.1 General ... 22
8.2.2 Spectrophotometer (optional) ... 22
8.2.3 Glass optical cuvette ... 22

9 Analytical procedure ... 22
9.1 General ... 22
9.2 Ion Chromatography method ... 22
9.2.1 General procedure ... 22
9.2.2 Interferences ... 23
9.2.3 Calibration ... 23
9.3 Thorin Method ... 24
9.3.1 Pre-treatment of sample solution before analysis for Thorin method ... 24
9.3.2 General procedure ... 24
9.3.3 Preparation of a chemical blank solution ... 25
9.3.4 Interferents ... 25

10 Determination of the characteristics of the method: sampling and analyse ... 26
10.1 General ... 26
10.2 Relevant performance characteristics of the method and performance criteria ... 26
10.2.1 General ... 26
10.2.2 Sampling procedure ... 26
10.2.3 Analyse procedure ... 27
10.3 Establishment of the uncertainty budget ... 28

11 Expression of results ... 29

12 Evaluation of the method in the field ... 31

13 Equivalence of Thorin and ion chromatography methods ... 31
13.1 General ... 31
13.2 Range ... 31
13.3 Matrix effect ... 31
13.4 Comparison of repeatability and trueness ... 31

14 Equivalence with an alternative method ... 31

15 Test report ... 32

Annex A (informative) Type of sampling equipment ... 33

Annex B (informative) Examples of absorbers ... 34

Annex C (informative) Example of assessment of compliance or reference method for SO2 with requirements on emission measurements ... 35

Annex D (informative) Evaluation of the method ... 43

Annex E (informative) Test of equivalency of Thorin method and ion chromatography ... 50

Annex ZA (informative) Relationship with EU Directives ... 60

Bibliography ... 61