

DIN EN 12868:2017-04 (E)

Child use and care articles - Method for determining the release of N-nitrosamines and N-nitrosatable substances from elastomer or rubber teats and soothers

Contents		Page
European foreword		5
Introduction		6
1	Scope	7
2	Normative references	7
3	Terms and definitions	7
4	Principle	8
5	Reagents	8
Table 1 -- Salts and their masses for 1 l of artificial saliva salt solution		9
6	Apparatus	9
7	Standard Solutions of N-nitrosamines	11
7.1	General	11
7.2	N-nitrosamines identified in teats	11
Table 2 -- Names, abbreviated names and CAS numbers of N-nitrosamines relevant for this standard, and the necessary limits of quantification		11
7.3	Calibration solutions (for detector response)	12
7.4	N-nitrosamine used as internal standard	12
8	Procedure	12
8.1	General	12
8.2	Sample Preparation	13
8.3	Sample A (for determination of N-nitrosatable substances)	14
8.3.1	Preparation and pre-boiling	14
8.3.2	Preparation of Migrate A (for determining N-nitrosatable substances) See A.2	14
8.3.3	Nitrosation of Migrate A (see A.3) and preparation of Solution A	15
8.4	Sample B (for determination of N-nitrosamines)	15
8.4.1	Preparation and pre-boiling	15
8.4.2	Preparation of Migrate B (for determining N-nitrosamines)	15
8.4.3	Preparation of Solution B	15
8.5	Preparation of extraction columns for Solutions A and B	15
8.6	Extraction of N-nitrosamines	16
8.6.1	From Solution A	16
8.6.2	From Solution B	16
8.7	Concentration of N-nitrosamines	16
8.7.1	In Extract A	16
8.7.2	In Extract B	17
8.8	Blank Test	17
8.9	Analysis	17
8.9.1	Calibration function	17
8.9.2	Determination of sample concentration	18
9	Calculation of results	18

9.1	General	18
9.2	Variability of results and calculation of means	19
9.2.1	Requirements for variability	19
9.2.2	Test for variability	19
9.2.3	Calculation of means	19
9.3	Amount of total N-nitrosatable substances migrating from Sample A, analysed and expressed as N-nitrosamines from Concentrate A	20
9.4	Amount of total N-nitrosamines migrating from Sample B, analysed and expressed as N-nitrosamines from Concentrate B	20
10	Confirmation of N-nitrosamines	20
11	Analytical tolerances	21
11.1	General	21
11.2	Analytical tolerances (see Annex F)	21
12	Compliance	21
13	Test report	21
Annex A (informative) Rationales		23
A.1	General	23
A.2	Migration Conditions (see 8.3.2)	23
A.3	Nitrosation conditions (see 8.3.3)	23
A.4	Weight of sample used (see 8.3.1)	23
A.5	Separate migrations (see 8.3.2 and 8.3.3)	24
A.6	Duplicate tests (see 8.1)	24
A.7	TEA and alternative detectors	24
A.8	Deviations (see 9.2, 9.3 and 11)	25
A.9	Main differences between this standard and EN 71-12 [3]	25
Annex B (informative) Suitable gas chromatographic method		26
Figure B.1 -- GC Chromatogram of a calibration solution recorded by TEA		27
Annex C (normative) Example of results calculation and test report		28
C.1	General	28
C.2	Example for variability testing and means calculation for analytical results	28
Table C.1 -- Example for variability testing and means calculation for Sample A		29
Table C.2 -- Example for variability testing and means calculation for Sample B		29
C.3	Results calculation and results table for test report	30
Table C.3 -- Example of Final results and their representation		31
C.4	Other Information for test report	31
Annex D (informative) Alternative Methods		32
D.1	General	32
D.2	Liquid Chromatography (LC)	32
Table D.1 -- Gradient profile for the given HPLC conditions		33
D.3	MS/MS conditions	33
Table D.2 -- Suitable MRM Transitions for MS/MS conditions		34
D.4	Confirmation and quantification of detected N-nitrosamines	34
Table D.3 -- Maximum permitted tolerances for relative ion intensities		34

Annex E (informative) Justification of an N-nitrosamine specific adjustment for NDiNA	36
Annex F (informative) Summary of the 2015 validation trial	38
F.1 Outline	38
Table F.1 -- Sample Descriptions for Validation Trial	38
F.2 Initial statistical analysis - N-nitrosatable substances	38
Table F.2 -- Mean data and calculations for total N-nitrosatable substances	38
F.3 Reproducibility Limit for N-nitrosatable substances	39
Table F.3 -- Overall means for total N-nitrosatable substances by method	39
Table F.4 -- Reproducibility limits for total N-nitrosatable substances by method	39
Table F.5 -- Reproducibility limits (mg/kg) for individual N-nitrosatable substances	40
F.4 Consideration of the NDiNA Reproducibility limit	40
F.5 Initial statistical analysis - N-nitrosamines	40
Table F.6 -- Mean data and calculations for total N-nitrosamines	40
F.6 Variability between Determinations	41
Table F.7 -- Summary of average repeatability limits for duplicate determinations of N- nitrosatable substances and N-nitrosamines	41
Table F.8 -- Methods used by laboratories (after removal of outliers)	42
Bibliography	44