DIN EN ISO 17678:2019-10 (E)

Milk and milk products - Determination of milk fat purity by gas chromato graphic analysis of triglycerides (ISO 17678:2019)

Contents

Page

European foreword ... 4

Foreword ... 5

1 Scope ... 7

2 Normative references ... 8

3 Terms and definitions ... 8

4 Principle ... 8

5 Reagents ... 9

6 Apparatus ... 9

7 Sampling ... 10

8 Procedure ... 11
8.1 Preparation of test samples ... 11
8.1.1 General ... 11
8.1.2 Isolation from butter or butteroil ... 11
8.1.3 Extraction according to the Röse-Gottlieb gravimetric method ... 11
8.1.4 Extraction from milk using silica gel columns ... 11
8.1.5 Extraction from cheese ... 12
8.2 Preparation of fat sample solution ... 12
8.3 Chromatographic triglyceride determination ... 12
8.3.1 Baseline drift ... 12
8.3.2 Injection technique ... 12
8.3.3 Calibration ... 12
8.3.4 Chromatographic conditions ... 13

9 Integration, evaluation and control of the analytical performance ... 14

10 Calculation and expression of results ... 16
10.1 Triglyceride composition ... 16
10.1.1 Calculation ... 16
10.1.2 Expression of test results ... 16
10.2 *S*-values ... 17
10.2.1 Calculation ... 17
10.2.2 Expression of test results ... 17
10.3 Detection of foreign fat ... 17

11 Precision ... 18
11.1 Interlaboratory test ... 18
11.2 Repeatability ... 18
11.3 Reproducibility ... 18

12 Test report ... 19

Annex A (normative) Preparation of the packed column ... 20
A.1 Reagents and materials ... 20
A.2 Apparatus ... 20

A.3 Silanization (deactivation of the glass surface) 20
A.4 Filling 22
A.5 Conditioning 22
Annex B (informative) Quantification of the foreign fat content 24
B.1 General 24
B.2 Calculation 24
B.3 Expression of test results 25
Annex C (informative) Uncertainty of measurement 26
Annex D (informative) Interlaboratory test 27
Bibliography 29

Figures

Figure 1 — Example of a triglyceride chromatogram of milk fat with baseline set correctly 15
Figure 2 — Example of a triglyceride chromatogram of milk fat with baseline set incorrectly 15
Figure A.1 — Silanization apparatus 21
Figure A.2 — Rinsing apparatus 21
Figure A.3 — Filling of the glass column 22

Tables

Table 1 — Coefficients of variation of triglyceride contents 16
Table 2 — *S*-limits for pure milk fats 18
Table 3 — Repeatability limits, *r*, for Formulae (3) to (7) 18
Table 4 — Reproducibility limits, *R*, for Formulae (3) to (7) 18
Table B.1 — 99 % Limits of detection of foreign fat added to milk fat as percentages 24
Table B.2 — S_f values of various foreign fats 25
Table C.1 — Extended S limits for pure milk fats including the expanded uncertainty 26
Table D.1 — Results of the interlaboratory test: Formula (3) 27
Table D.2 — Results of the interlaboratory test: Formula (4) 27
Table D.3 — Results of the interlaboratory test: Formula (5) 28
Table D.4 — Results of the interlaboratory test: Formula (6) 28
Table D.5 — Results of the interlaboratory test: Formula (7) 28