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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 22507-2008

**Animal and vegetable fats and oils -- Determination of the
content of trans fatty acid isomers of vegetable fats and oils --
Gas chromatographic method**

动植物油脂 植物油中反式脂肪酸异构体

(English Translation)

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1 Scope

This Standard specifies a gas chromatographic method using capillary columns for the determination of the content of trans fatty acid isomers of vegetable oils and fats.

The method is specially designed to evaluate, by a single capillary gas chromatographic (GC) procedure, the level of trans isomers as formed during (high temperature) refining, or during hydrogenation of vegetable oils or fats.

The method may also be used to report all other fatty acids (e.g. to obtain the full fatty acid composition and total amounts of saturated fatty acids, mono- unsaturated fatty acids and poly-unsaturated fatty acids) from the same sample and same analysis.

NOTE 1: The trans-isomer content as obtained with this method may not agree with the trans-

isomer content as obtained using other methods.

NOTE 2: During (high temperature) refining (deacidification and deodorization), only geometrical isomers are formed of the mono- and poly-unsaturated fatty acids; i.e. the double bond(s) remain(s) at the same natural position. During hydrogenation, both positional and geometrical isomers are formed.

NOTE 3: For some specific cis- and trans-isomers formed during hydrogenation, co-elution is possible. This could influence the accuracy of the result. The level of these isomers is usually negligible in normal partially hydrogenated oils and fats.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this

Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

GB/T 15687 Animal and vegetable fats and oils - Preparation of test sample (GB/T 15687-2008, ISO 661:2003, IDT)

GB/T 17376 Animal and vegetable fats and oils - Preparation of methyl esters of fatty acids (GB/T 17376-2008, ISO 5509:2000, IDT)

3 Terms and definitions

For the purposes of this Standard, the following terms and definitions apply.

3.1

content of trans fatty acid isomers of (high temperature) refined oils and fats

Sum of the C18:1 trans, C18:2 trans and C18:3 trans fatty acid methyl esters, expressed as a mass fraction of all fatty acid methyl esters.

3.2

content of trans fatty acid isomers of partially hydrogenated oils and fats

Sum of all trans double-bond-containing fatty acid methyl esters, expressed as a mass fraction of all fatty acid methyl esters.

NOTE: The content of trans fatty acid isomers is expressed in percent.

4 Principle

The methylated fatty acids of the sample are separated on a capillary gas chromatography column with a high polar stationary phase, with respect to their chain length, degree of (un)saturation and geometry and position of the double bonds.

5 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Carrier gas

preferentially helium or hydrogen, or otherwise nitrogen, of gas chromatographic quality, dried and with oxygen removed by suitable filters.

WARNING - Hydrogen, which is used only with capillary columns, can double the speed of the analysis (in comparison with helium) but is hazardous. Safety devices are available and it is essential that a suitable device be incorporated into the apparatus.

8 Preparation of test sample

Prepare the test sample in accordance with GB/T 15687.

Before taking the test portion from the sample, mix the sample thoroughly. Melt solid samples completely to ensure proper mixing

9 Preparation of methyl esters

Prepare the methyl esters from the triglycerides of the prepared test sample in accordance with GB/T 17376.

Methods specified in AOCS Official Method Ce2-66

[2] or IUPAC 2.301

[3] may also be used.

For trans-isomer determination in, for example, virgin olive oils, the trans- esterification routine as specified in GB/T 17376 is recommended to avoid any heating of the samples.

10.1 General

In conjunction with the analysis of the test sample (or a series of test samples), analyze a blank sample (n-heptane) and a reference sample of CRM (5.2).

10.2.1 Set up the gas chromatograph with one of the recommended

combinations of temperature and column as described in Table 1.

Measure the average carrier gas linear velocity as indicated in Table 1. with a split ratio of approximately 1:100.

See Annex B for typical chromatograms obtained with the conditions given in

Table 1.

The two C18:1 cis isomers should be clearly separated (see Annex B).

NOTE 2: Hydrogenated marine oils can give rise to a much wider range of trans isomers, making identification and quantification more difficult.

The C20:1 cis natural isomer should be positioned exactly amidst the last eluting trans isomer C18:3 (tcc) and the C18:3 ccc peak (linolenic acid) in (high temperature) refined oils.

If the separation is sufficient for this type of analysis, in (high temperature) refined oils there should be a small peak for the C18:1 trans isomer, two approximately equally sized C18:2 trans isomers, and four (sometimes five)

C18:3 trans isomers.

For partially hydrogenated oils and fats, the separation of the C18:1 13trans and the C18:1 9cis isomers should be visible on the chromatogram. This is required for an accurate peak-split between cis and trans isomers (see Annex

B).

The 18:1 13trans isomer always elutes with the 18:1 14trans isomer. Therefore, the peak for these two isomers should be identified as 18:1 (13+14) trans.

10.4 Peak identification

For (high temperature) refined oils and fats, the trans isomers are limited in number, as only geometrical isomers with the double bond(s) at the same natural position are formed. These specific isomers are for the C18 type of fatty acids: C18:1 9trans, C18:2 9c12t and C18:2 9t12c and for C18:3 the tct, cct, ctc and tcc 9.12.15 isomers (in some samples the C18:2 9t12t and C18:3 ttc isomers are found as well in very small amounts).

For partially hydrogenated oils and fats, the trans double-bond-containing isomers are identified using the equivalent chain length (ECL). For accurate peak identification with this system, the ECL values have to be determined after suitable calibration with available cis and trans fatty acid isomer standards

1 (see also Annex C).