RESOLUTION OENO 17/2000

INTERNATIONAL OENOLOGICAL CODEX

ANTI-FOAMING AGENTS (FATTY ACID MONO- AND DIGLYCERIDES)

SIN NO. 471

1. OBJECTIVE, ORIGIN AND SCOPE OF APPLICATION

The mixture of fatty acid glyceric mono- and diesters (with a small quantity of tri-esters), with fatty oils and acids and alimentary fats are termed mono- and diglycerides. The mixture of mono- and diglycerides used as anti-foaming agents are essentially constituted by oleic acid esters.

The product thus defined can contain small quantities of fatty acids and free glycerol. It is used under appropriate technological conditions and does not leave measurable traces in wine after filtering.

2. LABELING

The label must indicate the mono- and diglyceride content of the preparation, the storage and safety conditions, and the final date of use.

3. PROPERTIES

The product is usually found in the form of an oily liquid with a straw yellow color, a doughy product with an ivory color or a hard waxy solid with a white or off-white color. All of the forms have a pleasant odor and taste. The solid form can be found in flakes, powder or small granules.

The product used as an anti-foaming agent is liquid at normal temperatures, but can become cloudy at low temperatures

4. SOLUBILITY

Insoluble in water.

Soluble in ethanol, chloroform and benzene.

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5. IDENTIFYING CHARACTERISTICS

5.1. Hydrolysis of the Sample

Treat 1 g of the sample by reflux using a 0.5 M potassium hydroxide solution for 1 hour. Add 15 ml of water and acidify with hydrochloric acid diluted to 30 pp 100 (v/v) (R) (approximately 4-5 ml). Oily drops or a white/yellowish white precipitate will form. Extract the fatty acids released using 5 ml hexane, separating the solvent. Repeat the extraction with 5 ml of hexane and reunite the two extracts.

Set aside the aqueous phase.

5.2. Detection of the Fatty Acids in the Hexane Extract Using Gas Phase Chromatography

For the purpose of example, use may be made of a semi-polar column, e.g., Carbowax 20M ® measuring 25m x 0.32 mm x 0.25 μm phase thickness.

5.3. Detection of Glycerol

Place 5 ml of the aqueous phase in a test tube. Add an excess amount of powdered calcium hydroxide and place the test tube in boiling water for five minutes, stirring from time to time. Cool and filter.

Place one drop of the filtrate in a test tube and add approximately 50 mg of potassium hydrogen sulfate, At the end of the test tube, place a piece of filter paper soaked in the reagent obtained by mixing extemporaneously equal volumes of a sodium nitrosopentacyanoferrate solution (R) and piperidine (F'). Heat using a small flame. A blue coloring of the reactive paper indicates the presence of acrolein.

The color turns red by adding 1M sodium hydroxide solution.

6. TESTS

6.1. Drying Loss at 100 °C

Weigh exactly a quantity of about 5 g of the product to be analyzed in a glass crystallizing dish with a diameter of 70 mm, which has been preliminarily dried in an oven, cooled in a desiccator and calibrated. Place the crystallizing dish with the fatty material into a 103 °C oven and maintain this temperature for 30 minutes. Remove the crystallizing dish, let cooll in the desiccator, then weigh. Place the sample in the oven again for 30 minutes. Weigh it again after cooling. Drying loss in the oven is

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completed when weight loss does not exceed 0.05% per half-hour of heating.
Drying loss at 100 °C should be less than 2 pp 100.

6.2. Sulfur Ash

Sulfur ash is quantified as indicated in the Annex using a test sample of 5 g. The sulfur ash should weigh less than 0.2 g/kg.

6.3. Arsenic

Determined as indicated in the Annex using a test sample of 5 g. The arsenic should weigh less than 3 mg/kg.

6.4. Heavy metals

Test for heavy metals either:

- After mineralization at 450 ± 5 °C of the residue left by the drying loss test. Take up the ash using 1 ml of diluted hydrochloric acid (R) and one drop of concentrated nitric acid (R) while heating in a 100 °C water bath to activate dissolution, then decant in a 25 ml volumetric flask, washing the cap with distilled water. Fill up to gauge line.

Draw off a volume of v ml of solution corresponding to 2 g of the sample to be analyzed and proceed with the test for heavy metals as indicated in the Annex.

- or, after liquid mineralization of an sample weighed with precision to about 5 g using concentrated nitric acid (R), Perhydrol and a microwave digester to accelerate the operation.

Decant the liquid obtained in a 25 ml volumetric flask and fill to the line with the wash water. Continue as indicated in the heavy metal tests.
Heavy metal content, expressed in terms of lead, should be less than 10 mg/kg.

6.5. Lead

Using the technique set forth in the Compendium, determine the quantity of lead in one of the two aforementioned preparations (6.4). The lead content should be less than 5 mg/kg.

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6.6. Mercury

Using the technique described in the annex, determine the quantity of lead in one of the two aforementioned preparations (6.4). The lead content should be less than 1 mg/kg.

6.7. Cadmium

Using the technique detailed in the annex, determine the quantity of cadmium in one of the two aforementioned preparations (6.4). The lead content should be less than 1 mg/kg.

6.8. Free Fatty Acids

Prepare 125 ml of a mixture of equal volumes of isopropyl alcohol and toluene. Add 2 ml of 1 pp 100 phenolphthalein solution (m/v) in isopropyl alcohol and neutralize using an alkaline solution until a persistent but weak pink coloring appears.

Weigh with precision an amount of approximately 5 g of the sample to be analyzed in a 500 ml conical flask. Add the neutralized solvent mixture and dissolve the test sample, by heating if necessary, while stirring vigorously. Pour the 0.1 M potassium hydroxide solution until a pink color identical to that obtained during the solvent neutralization process is obtained. Let n be the volume in ml poured:

Fatty acid content expressed in g of oleic acid pp 100 (m/m):

- 2.8n / test sample in g

The fatty acid content in terms of oleic acid should be less than 3 pp 100 (m/m).

6.9. Soaps

Weigh precisely about 10 g of the product to be analyzed in a 250 ml conical flask. Add a mixture of 60 ml of acetone and 0.15 ml of 0.5 pp 100 (m/v) bromophenol blue solution in 95% alcohol by volume which has first been neutralized with a 0.1 M hydrochloric acid solution or a 0.1 M sodium hydroxide solution. Gently heat in a 70 °C water bath and titrate with a 0.1 M hydrochloric acid solution until the blue color disappears. Let sit for 20 minutes. Heat until the precipitate redissolves and, if the blue color reappears, continue titration.

1 ml 0.1 M hydrochloric acid solution corresponds to 0.0304 g of sodium oleate ($NaC_{18}H_{33}O_2$).

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Soap content expressed in g of sodium oleate pp 100 (m/m):

- 3.04n / test sample in g

Soap content expressed in g of sodium oleate should be less than 6 pp 100 (m/m).

6.10. Monoglycerides

6.10.1. Sample preparation

If the sample is in solid form, melt it by heating it to its melting point at a temperature of less than 10 °C. Liquid samples which are cloudy or have particles in them should also be heated. Mix vigorously.

6.10.2. Method

Weigh precisely a test sample, Q, of approximately 1 g to be analyzed in a 100 ml cylindrical flask. Dissolve using 25 ml of chloroform. Transfer this solution to a decanting glass. Wash the cylindrical flask with 25 ml of chloroform, then with 25 ml of water and add these liquids to the contents of the decanting glass.

Seal the decanting glass hermetically. Stir for 30-60 seconds. Let the two phases separate out (add 1-2 ml of crystallizable acetic acid (R) to break the emulsion). Collect the aqueous phase in a 500 ml conical flask with an emery stopper. Extract the chloroform phase remaining in the decanting glass twice with 25 ml of water. Separate the aqueous phase and place it in the 500 ml conical flask. These aqueous extracts will be used for the free glycerol analysis.

Transfer the chloroform from the decanting glass to a 500 ml conical flask with an emery stopper. Add 50 ml of periodic acetic acid solution (R) while stirring.

In the two other 500 ml conical flasks with emery stoppers to be used as "blanks", place 50 ml of chloroform and 10 ml of water. Add 50 ml of periodic acetic acid solution (R) while stirring to each of the two flasks. Let the three flasks sit at least 30 minutes, but no more than 90 minutes.

While gently stirring, add 20 ml of potassium iodide solution (R) to each of these containers. Let sit at least 1 minute but no more than 5 minutes before volumetric analysis.

Add 100 ml water and titrate with a 0.05 M sodium thiosulfate solution using a magnetic stirrer until the brown color disappears from the aqueous phase. Add 2 ml of starch solution (R) and continue to add the reagent until the iodine disappears from the chloroform layer and the blue color disappears from the aqueous phase.

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6.10.3. Calculate the percentage of monoglycerides using the formula:

- (B-S) . M . 17.927 / P

B is the average volume in ml of the sodium thiosulfate solution used for analysis of the "blanks" containing chloroform.

S is the amount of sodium thiosulfate solution in ml used to titrate the sample.

M is the exact molarity of the sodium thiosulfate solution.

P is the weight of the sample to be analyzed in the volume of chloroform used for the analysis.

17.927 is the molar mass of glycerol monostearate, divided by 20.

The monoglyceride content expressed in terms of glycerol monostearate should be greater than 30 pp 100 (m/m).

6.11. Free glycerol

Add 50 ml of periodic acetic acid solution (R) to the aqueous extracts obtained during the monoglyceride-analysis process. Simultaneously prepare a "blank" by adding to 75 ml of water in a 500 ml conical flask 50 ml of periodic acetic acid solution (R). Continue the determination process as indicated in the method described for monoglycerides.

Calculate the percentage of glycerol using the following formula:

- (b-S)M . 2.30 / Q

b is the volume in ml of sodium thiosulfate solution used in the quantitative analysis the "blank" containing 75 ml of water

S is the volume in ml of sodium thiosulfate solution used in the quantitative analysis of the aqueous extracts

M is the molarity of the sodium thiosulfate solution.

Q is the weight of the first sample to to be analyzed (see monoglyceride determination).

Glycerol content should be less than 7 pp 100 (m/m).

N.B. : Glycerol can also be disclosed and identified by high performance liquid chromatography (HPLC) (5.3).

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7. STORAGE

Anti-foaming agents should be kept in completely water-tight containers and away from heat.

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