Designation: E1346 – 16

Standard Practice for Bulk Sampling, Handling, and Preparing Edible Vegetable Oils for Sensory Evaluation[1]

This standard is issued under the fixed designation E1346; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the recommended procedures for bulk sampling, handling, and preparing edible vegetable oil (liquid at room temperature) prior to sensory evaluation.

1.2 This practice is consistent with the background information presented in ASTM STP 433, ASTM Manual 26, and ASTM STP 758. These should be consulted for supplemental guidance.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Publications:*[2]

ASTM STP 433 Basic Principles of Sensory Evaluation

ASTM Manual 26 Manual on Sensory Testing Methods, 2nd Ed.

ASTM STP 758 Guidelines for the Selection and Training of Sensory Panel Members

2.2 *AOCS Standard:*[3]

Method C1-47 Sampling

3. Summary of Practice

3.1 This practice consists of the following basic steps: removing oil from bulk source, transporting and storing oil prior to evaluation, preparing oils for evaluation, presenting samples to panel, and cleaning glassware.

4. Significance and Use

4.1 This practice is designed for use by the oil processor or research laboratory for evaluation by a trained sensory panel, or for use by quality control (QC) and quality assurance (QA) personnel for sampling from a tank truck, car, or any other bulk transportation container, or by both.

4.2 The consistent use of this practice will provide representative samples for all sensory, chemical and physical analyses and will protect the oil from oxidation.

4.3 The objective of this practice is to ensure that the sample is representative of the sample source from the time of sampling until the time of evaluation and to protect oil quality during that time.

4.4 This practice addresses neither evaluation and scaling techniques, nor the sampling, handling, and preparing of solid fats.

5. Apparatus

5.1 *Liquid Zone Sampler,* or core sampler, or trier.[4]

5.2 *Wide-Mouth Jars* made of polyethylene terephthalate, 0.5 to 1.0 L.

5.3 *Amber Glass Bottles,* 250 mL to 1 L, with narrow-mouth tops that will withstand freezer temperatures.

5.4 *Plastic Caps with polytetrafluoroethylene (PTFE) Liners,* or tape (PTFE pipe thread tape), to cover top of bottle opening before capping with new non-metallic screw type caps. Tape should be 2.5 cm in width or wider to completely cover bottle openings.

5.5 *Glass Funnels.*

5.6 *Glove Box* with inert gas (for example, nitrogen) atmosphere, including an oxygen scavenging device.

5.7 *Glass Vial,* 50 mL. Use amber glass for odor and flavor evaluation; and clear glass for visual examination of oil.

5.8 *Standard Disposable Glass Pipets,* 10 mL, one per each sample.

[1] This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluationand is the direct responsibility of Subcommittee E18.06 on Food and Beverage Evaluation.

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[2] Available from ASTM International Headquarters, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.

[3] Available from American Oil Chemists' Society, P.O. Box 3989, Champaign, IL 61826.

[4] Available from Refinery Supply Co., Tulsa, OK.

5.9 *Circulating Water bath,* with automatic timer, thermostat and rack.

5.10 *Water bath Thermometer,* with range from 20 to 100°C in 1°C divisions, calibrated for 76 mm immersion, 305 mm long.

6. Precautions

6.1 Oil submitted for chemical and physical testing and for sensory evaluation should be from the same bulk sampling. Tank trucks, cars, or any other bulk transportation containers may be filled with as many as seven layers and each level of oil may be slightly different in quality. Oil samples should be handled in the same manner and time frame to ensure high data correlation.

6.2 Obtain a representative oil sample for all evaluations (sensory, chemical, instrumental); unblended multiple samples may produce different results.

6.3 Do not allow glass containers in processing or production areas where oil sampling is done.

6.4 Use only new, clean, dry, and odor-free polyethylene terephthalate (PET) wide-mouth jars to collect oil samples; dispose of jars rather than cleaning them.

6.5 Store oil in amber glass bottles to protect the oil from light oxidation.

6.6 Choose size of storage bottle based on purpose of evaluation, amount of oil required for each testing session or for number of panelists, and amount of oil needed for instrumental or chemical tests. For example, a 1 L sample of oil that requires evaluation quarterly should be stored in four 250-mL bottles.

6.7 Use PTFE-lined caps or PTFE tape under caps to protect oil from off-odors or flavors imparted from metallic or unlined plastic caps.

6.8 Do not expose oil to any environmental condition (for example, ligh, heat, oxygen, moisture) or any equipment (metals) that will cause oxidation of the oil and alter sensory characteristics of the oil.

6.9 Transfer oil from plastic bottle to recommended glass bottles within one hour of collection and flush headspace with inert gas to minimize potential transfer of odors or flavors from the plastic container to the oil (conduct procedure in glove box under nitrogen atmosphere).

6.10 Keep an inert gas in contact with the oil at all times to avoid exposure of the oil to oxygen.

6.11 Discard any unused oil.

7. Procedures for Handling Samples Obtained from Bulk Storage

7.1 Refer to the AOCS Official Method C1-47 on oil sampling for specifications for detailed information on equipment and procedures.

7.2 Steps 7.3 – 7.5 should be conducted in a glove box under inert gas atmosphere.

7.3 Flush bottle with inert gas prior to filling the bottle.

7.4 Fill bottle with oil, leaving 0.5 to 1 cm of headspace between the oil and the cap liner.

7.5 Flush headspace with inert gas to remove all oxygen which deteriorates the oil, and cap bottle.

7.5.1 Flush only the headspace with inert gas since bubbling nitrogen through oil for short periods of time has little benefit.

7.5.2 Analyze headspace for oxygen to ensure that bottles are being flushed correctly as follows:

7.5.2.1 Flush headspace of bottle with inert gas, seal with silicone rubber septum in screw type cap.

7.5.2.2 Withdraw a gas sample with a syringe through the septum.

7.5.2.3 Inject sample into gas chromatograph with thermal-conductivity detector using a two column system. Column conditions are: ethylvinyl benzene-divinylbenzene polymer 80 to 100 mesh (3 ft by ⅛ in.) and molecular sieve 5A 80 to 100 mesh (9 ft by 1⁄16 in.) with 25°C oven temperature and 20 mL/min helium flow rate.[5]

7.6 Store all oils at -20 or 5°C, except for the sample for initial evaluation, which may be held at ambient temperature (25°C) in the dark for 1 h after sampling from bulk storage before analyses.

7.7 Samples should be held a maximum of 2 days at 5 ± 2°C in the dark before evaluation. If evaluation is not possible within this time frame, filled containers should be held at -20°C. Always store samples in the dark.

7.8 Do not open bottles until ready for sample evaluation. During this holding period, bottles should remain sealed with inert gas in the headspace.

7.9 *Winterized Oil:*

7.9.1 Frozen sample is removed from cold storage and held at refrigerated (5 ± 2°C) temperature until completely homogeneous, that is, clear, with no visible solids. The time requirements for thawing the oil will vary depending upon container size.

7.9.2 Sample must be mixed just prior to evaluation by inverting bottle several times to ensure homogeneity and to minimize potential density differences within the container; for example, a 500 mL bottle with between 0.5 and 1 cm headspace is inverted ten times.

7.10 *Non-Winterized Oil:*

7.10.1 The frozen sample is removed from cold storage and held at refrigerated (5 ± 2°C) temperature until it stabilizes at that temperature (5°C). Next, move container to ambient temperature (25 ± 5°C) until completely homogeneous; clear, no visible solids.

7.10.2 Sample must be mixed just prior to evaluation by inverting bottle several times to ensure homogeneity and to minimize potential density differences within the container; for example, a 500 mL bottle with between 0.5 and 1 cm headspace is inverted ten times.

[5] Saguy, I., Goldman, M., and Karel, M., "Prediction of Beta-Carotene Decolorization in Model System Under Static and Dynamic Conditions of Reduced Oxygen Environment," *Journal of Food Science*, Vol 50, 1985, pp. 526–530.

7.11 Samples for instrumental, chemical or physical testing should be taken from the thawed sample just prior to sensory testing. All evaluations should be conducted within the same time frame as the sensory tests to ensure valid test results for correlation analyses. Oil that cannot be tested immediately must have the headspace thoroughly re-flushed with inert gas and the bottle recapped with PFTE tape or a PFTE-lined plastic cap and stored at 5°C for no longer than 12 h. Any leftover sample should be discarded. Re-freezing is abusive to the oil and is not recommended.

7.12 Do not thaw samples by heating above the ambient temperature (25°C) by microwave or in water bath. These procedures are abusive and may deteriorate the oil and develop off-odors and flavors, causing the sample to be less than representative of the sample source.

7.13 For winterized oil, once the oil has reached refrigerated temperature (5°C), it must be evaluated within 12 h if the bottle has been opened, or within 2 days if the bottle has not been opened.

7.14 All samples must be at ambient temperature at the time of preparation for sensory evaluation.

8. Procedures for Preparation of Oils for Sensory Evaluation

8.1 Visual evaluations of oils should be conducted separately from odor and flavor testing. Place oils in clear glass vials. Do not heat oil samples.

8.2 Fill water bath with distilled water and heat to 50°C.

8.3 Pipet 10 mL oil into each glass vial and cap. Do not allow drips on inside of container as it will increase the surface area of the oil to oxygen and cause subsequent deterioration.

8.4 Immerse sample vials into pre-heated water bath at a level sufficient to cover the oil in the vial. Vials should be suspended in the water bath on racks rather than sitting on the bottom to allow an even flow of water around each vial and to ensure that the temperature is constant without hot or cold spots. Do not cover the water bath. Vials are spaced equidistant from each other and the edges of the water bath. No vial should touch another vial or the side of the water bath. The water bath must be of sufficient size to hold all sample vials required for a single panel.

8.5 Maintain vials in the controlled temperature, pre-heated water bath for sufficient time for oils to reach serving temperature of 50 ± 1°C. Determine minimum time required to bring oil to serving temperature, (usually 6 to 10 min). Serve sample immediately. Sample should not be held longer than 30 min since oil exposure to elevated temperature and oxygen deteriorate oil quality.

8.6 If more than one water bath must be used, attention should be given to standardize the number and placement of vials within each bath. If the number of vials required exceeds the capacity of a single water bath, each judge's sample set should be placed in the same water bath. Duplicate water baths must be equivalent.

8.7 *Sample Presentation:*

8.7.1 When ready to serve oils, remove vials from water bath, wipe residual water from vials and serve oils immediately. If appropriate to the test, present samples monadically when possible to keep oils at proper evaluation temperature (50 ± 1°C) and provide a timed minimum interval between samples for clearing the palate.

8.7.2 If samples are presented in pairs or other multiples, it is recommended that a method such as aluminum blocks be used to maintain uniform sample temperature in the booth. Aluminum blocks heated to a temperature of 5°C higher than the serving temperature of the oil will keep the sample at the proper serving temperature for 10 min. Blocks should have openings for vials that can extend 4 cm above the top of the oil in the vial. The diameter of the opening should be a maximum of 1 cm wider than the vial to allow adequate transfer of heat.

8.8 *Special Concerns:*

8.8.1 PFTE-lined caps or PFTE tape applied over the bottle opening under the caps are recommended as least likely to affect headspace volatiles or the oil's sensory characteristics during heating.

8.8.2 Amber glass is recommended for odor and flavor evaluation only, (not visual), however, colored lighting such as red fluorescent bulbs, low sodium lighting, or theatrical gels and filters can also be effective.

9. Glassware

9.1 Use new, clean glassware for each evaluation. If this is not economically practical, ensure that glassware is clean and odor-free prior to each use.

9.2 Clean vials after each use by washing with commercial, unscented, glassware-washing detergent to remove all oil residue.

9.3 Test both glass vials and evaluation glassware for cleanliness by rinsing with distilled water, (water should sheet off surface rather than form droplets). If droplets form, clean glassware with alcoholic NaOH (sodium hydroxide), rinse with distilled water, and re-evaluate for sheeting. Discard any glassware that does not sheet clean after the NaOH treatment.

9.3.1 Prepare alcoholic NaOH as follows: dissolve 120 g NaOH in 120 mL H_2O and dilute to 1 L with isopropyl alcohol. Soak glassware for no more than 30 min to minimize etching.

9.4 Discard caps and liners after one use since it is not possible to sufficiently clean these for reuse.

9.5 Store glassware in closed cabinet away from chemical odors to protect from contamination.

9.6 Discard glassware that is etched or that does not meet minimum standards for cleanliness.