Food and Drug Administration, HHS

§ 172.250 Petroleum naphtha.

Petroleum naphtha may be safely used in food in accordance with the following conditions:
(a) It is used as the salt(s) of one or more of the fatty acids meeting the requirements of §172.860, as a component of protective coatings applied to fresh fruits and vegetables.
(b) It is used at a level not in excess of that reasonably required to produce its intended effect.

General Instructions

All glassware should be scrupulously cleaned to remove all organic matter such as oil, grease, detergent residues, etc. Examine all glassware, including stoppers and stopcocks, under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure, it is recommended practice to rinse all glassware with purified isoctane immediately before use. No grease is to be used on stopcocks or joints. Great care to avoid contamination of petroleum naphtha samples in handling and to assure absence of any extraneous material arising from inadequate packaging is essential. Because some of the polynuclear hydrocarbons sought in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

Analytical Specification for Petroleum Naphtha

<table>
<thead>
<tr>
<th>Wavelength (mill-microns)</th>
<th>Maximum absorbance per centimeter optical pathlength</th>
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<tbody>
<tr>
<td>280–289</td>
<td>0.15</td>
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<tr>
<td>290–299</td>
<td>0.13</td>
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<tr>
<td>300–359</td>
<td>0.08</td>
</tr>
<tr>
<td>360–400</td>
<td>0.02</td>
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</tbody>
</table>

Reagents

Isooctane (2,2,4-trimethylpentane). Use 180 milliliters in a 250-milliliter Erlenmeyer flask, add 1 milliliter of purified n-hexadecane, insert the head assembly, allow nitrogen gas to flow into the inlet tube and connect the outlet tube to a solvent trap and vacuum line in such a way as to prevent any back flow of condensate into the flask. The

APPARATUS

Separatory funnels, 250-milliliter, and 2,000-milliliter capacity, equipped with tetrafluoroethylene polymer stopcocks.
Erlenmeyer flask, 125-milliliter with 24/40 standard taper neck.
Evaporation flask, 250-milliliter capacity all glass flask equipped with 24/40 standard taper stopper having inlet and outlet tubes to permit passage of nitrogen across the surface of the container liquid to be evaporated.
Condenser, 24/40 joints, fitted with drying tube, length optional.
Spectrophotometric cells. Fused quartz cells, optical path length in the range of 5,000 centimeters ± 0.005 centimeter; also for checking spectrophotometer performance only, optical path length in the range 1,000 centimeter ± 0.005 centimeter. With distilled water in the cells, determine any absorbance difference.
Spectrophotometer. Spectral range 250–400 mμ with spectral slit width of 2 mμ or less; under instrument operating conditions for these absorbance measurements, the spectrophotometer shall also meet the following performance requirements:
Absorbance repeatability, ± 0.01 at 0.4 absorbance.
Absorbance accuracy, ± 0.05 at 0.4 absorbance.
Wavelength repeatability, ± 0.2 millimicron.
Wavelength accuracy, ± 1.0 millimicron.
Ultraviolet lamp. Long wavelength (3400–3800 Å).

1As determined by procedure using potassium chromate for reference standard and described in National Bureau of Standards Circular 484, Spectrophotometry, U.S. Department of Commerce, (1949). The accuracy is to be determined by comparison with the standard values at 290, 345, and 400 millimicrons. The procedure is incorporated by reference. Copies of the material incorporated by reference are available from the Center for Food Safety and Applied Nutrition (HFS–200), Food and Drug Administration, 5100 Paint Branch Pkwy., College Park, MD 20740, or available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6039, or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.
PROCEDURE

Determination of ultraviolet absorbance. Add a 25-milliliter aliquot of the hydrocarbon solvent together with 1 milliliter of hexadecane to the 125-milliliter Erlenmeyer flask. While flushing with nitrogen, evaporate to 1 milliliter on a steam bath. Nitrogen is admitted through a 8-millimeter outer-diameter tube, drawn out into a 281-centimeter long and 1.5-millimeter inner-diameter capillary tip. This is positioned so that the capillary tip extends 4 centimeters into the flask. The nitrogen flow rate is such that the surface of the liquid is barely disturbed. After the volume is reduced to that of the 1 milliliter of hexadecane, the flask is left on the steam bath for 10 more minutes before removing. Add 10 milliliters of purified isooctane to the flask and reevaporate the solution to a 1-milliliter volume in the same manner as described above, except do not heat for an added 10 minutes. Repeat this operation twice more. Let the flask cool.

Add 10 milliliters of methyl alcohol and about 0.5 gram of sodium borohydride. (Minimize exposure of the borohydride to the atmosphere; a measuring dipper may be used.) Immediately fit a water-cooled condenser equipped with a 24/40 joint and with a drying tube into the flask, mix until the sodium borohydride is dissolved, and allow to stand for 30 minutes at room temperature, with intermittent swirling. At the end of this time, disconnect the flask and evaporate the methyl alcohol on the steam bath under nitrogen until sodium borohydride begins to drop out of solution. Remove the flask and let it cool.

Add 6 milliliters of isooctane to the flask and swirl to wash the crystalline slurry. Carefully transfer the isooctane extract to a 250-milliliter separatory funnel. Dissolve the crystals in the flask with about 25 milliliters of distilled water and pour this also into the separatory funnel. Adjust the water volume in the separatory funnel to about 100 milliliters and shake for 1 minute. After separation of the layers, draw off the aqueous layer into a second 250-milliliter separatory funnel. Transfer the hydrocarbon layer in the first funnel to a 25-milliliter volumetric flask.

Carefully wash the Erlenmeyer flask with an additional 6 milliliters of isooctane, swirl, and transfer to the second separatory funnel. Shake the funnel for 1 minute. After separation of the layers, draw off the aqueous layer into a second 250-milliliter separatory funnel. Transfer the isooctane in the second funnel to the volumetric flask. Again wash the Erlenmeyer flask with an additional 6 milliliters of isooctane, swirl, and transfer to the first separatory funnel. Shake the funnel for 1 minute. After separation of the layers, draw off the aqueous layer and discard. Transfer the isooctane layer to the volumetric flask and adjust the volume to 25 milliliters of isooctane. Mix the contents well, then transfer to the first separatory funnel and wash twice with 50-milliliter portions of distilled water. Discard the aqueous layers after each wash.

Determine the ultraviolet absorbance of the isooctane extract in 5-centimeter path length cells compared to isooctane as reference between 280–400 μ. Determine a reagent blank concurrently with the sample, using 25 milliliters of purified isooctane instead of a solvent sample and measuring the ultraviolet absorbance of the blank between 280–400 μ.

The reagent blank absorbance should not exceed 0.04 per centimeter path length between 280–289 μ; 0.020 between 290–359 μ; and 0.010 between 360–400 μ.

Determination of boiling-point range. Use ASTM method D86–82, “Standard Method for Distillation of Petroleum Products,” which is incorporated by reference. Copies may be obtained from the American Society for Testing Materials, 100 Barr Harbor Dr., West Conshohocken, Philadelphia, PA 19428-2959, or may be examined at the National Archives and Records Administration (NARA).

For information on the availability of this material at NARA, call 202-741-6030, or go to:
http://www.archives.gov/federal_register/
§ 172.275 Synthetic paraffin and succinic derivatives.

Synthetic paraffin and succinic derivatives identified in this section may be safely used in food, subject to the following restrictions:

(1) Not less than 90 percent butyl oleate.

(2) Not more than 1.5 percent unsaponifiable matter.

(b) The additive is used or intended for use at a level not to exceed 2 percent by weight in an aqueous emulsion in dehydrating grapes to produce raisins, whereby the residue of the additive on the raisins does not exceed 100 parts per million.

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§ 172.270 Sulfated butyl oleate.

Sulfate butyl oleate may be safely used in food, subject to the following prescribed conditions:

(a) The additive is prepared by sulfation, using concentrated sulfuric acid, of a mixture of butyl esters produced by transesterification of an edible vegetable oil using 1-butanol. Following sulfation, the reaction mixture is washed with water and neutralized with aqueous sodium or potassium hydroxide. Prior to sulfation, the butyl oleate reaction mixture meets the following specifications:

(1) Not less than 90 percent butyl oleate.

(2) Not more than 1.5 percent unsaponifiable matter.

(b) The additive is used or intended for use at a level not to exceed 2 percent by weight in an aqueous emulsion in dehydrating grapes to produce raisins, whereby the residue of the additive on the raisins does not exceed 100 parts per million.

[57 FR 12711, Apr. 13, 1992]