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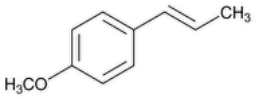
Anethole



$C_{10}H_{12}O$ 148.20

Benzene, 1-methoxy-4-(1-propenyl)-, (*E*)-;

(*E*)-*p*-Propenylanisole CAS RN®: 4180-23-8.

Synthetic CAS RN®: 104-46-1.

Change to read:

DEFINITION

Anethole is obtained from Anise Oil and other sources, or it is prepared synthetically. It contains NLT 98.0% and NMT 102.0% of (*E*)-1-▲methoxy▲ (ERR 1-Nov-2022) -4-(1-propenyl) benzene (or *trans*-anethole) ($C_{10}H_{12}O$).

IDENTIFICATION

- **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197F**
- **B. Chromatographic Identity**

Analysis: Proceed as directed in the *Assay for trans-Anethole*.

Acceptance criteria: The retention time of the major peak, excluding the internal standard peak, of the *Sample solution* corresponds to the anethole peak of the *Standard solution*.

ASSAY

- **Assay for *trans*-Anethole**

Internal standard solution: 2 mg/mL of USP Menthol RS (internal standard) in hexanes

Standard solution: 2 mg/mL of USP Anethole RS in *Internal standard solution* (use a freshly prepared solution)

Sample solution: 2 mg/mL of Anethole in *Internal standard solution* (use a freshly prepared solution)

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.53-mm × 30-m capillary; bonded with a 1.0-µm layer of phase G16

Temperatures

Injection port: 250°

Detector: 250°

Column: 125° (isothermally)

Carrier gas: Helium

Flow rate: 10 mL/min

Injection volume: 1.0 µL

Injection type: Split injection; split ratio of 10:1

Run time: 30 min

System suitability

Sample: *Standard solution*

[Note—The relative retention times for menthol and *trans*-anethole are about 0.5 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 15 between menthol and *trans*-anethole

Tailing factor: 0.8–2.0 for menthol and *trans*-anethole peaks

Relative standard deviation: NMT 2.0% for the peak response ratio of *trans*-anethole to the internal standard

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of *trans*-anethole ($C_{10}H_{12}O$) in the portion of Anethole taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of *trans*-anethole to the internal standard (peak response of *trans*-anethole/peak response of the internal standard) from the *Sample solution*

R_S = peak response ratio of *trans*-anethole to the internal standard (peak response of *trans*-anethole/peak response of the internal standard) from the *Standard solution*

C_S = concentration of USP Anethole RS in the *Standard solution* (mg/mL)

C_U = concentration of Anethole in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0%

IMPURITIES

• **Limit of *cis*-Anethole, *p*-Anisaldehyde, and Other Volatile Impurities**

Internal standard solution: 0.01 mg/mL of USP Menthol RS (internal standard) in hexanes

System suitability solution: 4 mg/mL of USP Anethole RS in *Internal standard solution* after exposure to UV light for 1 h

Standard solution: 0.01 mg/mL of USP Anethole RS and 0.01 mg/mL of USP p-Anisaldehyde RS in *Internal standard solution* (use a freshly prepared solution)

Sample solution: 2 mg/mL of Anethole in *Internal standard solution* (use a freshly prepared solution)

Chromatographic system: Proceed as directed in the *Assay*, except use a *Run time* of 60 min.

System suitability

Samples: *System suitability solution* and *Standard solution*

[Note—See *Table 1* for the relative retention times.]

Table 1

Name	Relative Retention Time
Menthol (internal standard)	0.5
cis-Anethole	0.75
trans-Anethole	1.0
p-Anisaldehyde	2.2

Suitability requirements

Resolution: NLT 5 between *cis*-anethole and *trans*-anethole, *System suitability solution*

Relative standard deviation: NMT 5.0% for the peak response ratio of *trans*-anethole to the internal standard and NMT 5.0% for the peak response ratio of *p*-anisaldehyde to the internal standard, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of *p*-anisaldehyde in the portion of Anethole taken:

$$\text{Result} = (R_{U1}/R_{S1}) \times (C_{S1}/C_U) \times 100$$

R_{U1} = peak response ratio of *p*-anisaldehyde to the internal standard (peak response of *p*-anisaldehyde/peak response of the internal standard) from the *Sample solution*

R_{S1} = peak response ratio of *p*-anisaldehyde to the internal standard (peak response of *p*-anisaldehyde/peak response of the internal standard) from the *Standard solution*

C_{S1} = concentration of USP p-Anisaldehyde RS in the *Standard solution* (mg/mL)

C_U = concentration of Anethole in the *Sample solution* (mg/mL)

Calculate the percentage of *cis*-anethole or any other unspecified volatile impurity in the portion of Anethole taken:

$$\text{Result} = (R_{U2}/R_{S2}) \times (C_{S2}/C_U) \times 100$$

R_{U2} = peak response ratio of *cis*-anethole or any other unspecified impurity to the internal standard (peak response of *cis*-anethole or any other unspecified impurity/peak response of the internal standard) from the *Sample solution*

R_{S2} = peak response ratio of *trans*-anethole to the internal standard (peak response of *trans*-anethole/peak response of the internal standard) from the *Standard solution*

C_{S2} = concentration of USP Anethole RS in the *Standard solution* (mg/mL)

C_U = concentration of Anethole in the *Sample solution* (mg/mL)

Acceptance criteria: Disregard peaks that are less than 0.05% for any unspecified impurities and any peaks due to solvent.

Total impurities including *cis*-anethole, *p*-anisaldehyde, and all other unspecified impurities: NMT 2.0%

• **Limit of Phenols**

Sample: 1 mL

Analysis: Shake the *Sample* with 20 mL of water, and allow the liquids to separate. Pass the water layer through a filter paper previously moistened with water, and to 10 mL of the filtrate add 3 drops of ferric chloride TS.

Acceptance criteria: No purple or purplish color is produced.

SPECIFIC TESTS

• **Specific Gravity ⟨841⟩:** 0.983–0.988

• **Distilling Range,** *Method I* **⟨721⟩:** 231°–237°, a correction factor of 0.063°/mm being applied as necessary

• **Optical Rotation,** *Angular Rotation* **⟨781A⟩:** −0.15° to +0.15°

• **Refractive Index ⟨831⟩:** 1.557–1.561

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers.

• **Labeling:** Label to indicate whether it is of natural sources or is prepared synthetically.

• **USP Reference Standards ⟨11⟩**

USP Anethole RS

USP p-Anisaldehyde RS

USP Menthol RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ANETHOLE	Documentary Standards Support	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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