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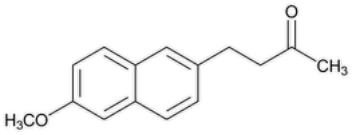
Nabumetone



$C_{15}H_{16}O_2$ 228.29

2-Butanone, 4-(6-methoxy-2-naphthalenyl)-;

4-(6-Methoxy-2-naphthyl)-2-butanone CAS RN®: 42924-53-8; UNII: LW0TIW155Z.

DEFINITION

Nabumetone contains NLT 98.0% and NMT 101.0% of nabumetone ($C_{15}H_{16}O_2$), calculated on the anhydrous basis.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests** ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020)

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Solution A: Water and glacial acetic acid (999:1), filtered and degassed

Solution B: Acetonitrile and tetrahydrofuran (700:300), filtered and degassed

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	60	40
12	60	40
28	20	80
29	60	40
30	60	40

Standard solution: 1.0 mg/mL of USP Nabumetone RS in acetonitrile

Sample solution: 1.0 mg/mL of Nabumetone in acetonitrile

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 15-cm; 4-µm packing L1

Flow rate: 1.3 mL/min

Injection volume: 10 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of nabumetone ($C_{15}H_{16}O_2$) in the portion of Nabumetone taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

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

Acceptance criteria: 98.0%–101.0% on the anhydrous basis

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.1%
- **Organic Impurities**

Solution A, Solution B, Mobile phase, and **Chromatographic system:** Proceed as directed in the *Assay*.

System suitability solution: 1 mg/mL of USP Nabumetone RS and 1 µg/mL of USP Nabumetone Related Compound A RS in acetonitrile

Sample solution: Use the *Sample solution* from the *Assay*.

System suitability

Sample: *System suitability solution*

[Note—The relative retention times are about 0.9 for nabumetone related compound A and 1.0 for nabumetone; see *Table 2*.]

Suitability requirements

Resolution: NLT 1.5 between nabumetone related compound A and nabumetone

Column efficiency: NLT 3600 theoretical plates

Tailing factor: 0.8–2.0 for the nabumetone peak

Relative standard deviation: NMT 2.0%

Analysis

Sample: *Sample solution*

Calculate the percentage of each impurity in the portion of Nabumetone taken:

$$\text{Result} = (F \times r_U)/\{r_N + [\Sigma(F \times r_U)]\} \times 100$$

F = relative response factor for each impurity (see *Table 2*)

r_U = peak area of each impurity from the *Sample solution*

r_N = peak area of nabumetone from the *Sample solution*

Acceptance criteria: See *Table 2*.

Table 2

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (w/w, %)
6-Methoxy-2-naphthaldehyde	0.73	0.12	0.1
4-(6'-Methoxy-2'-naphthyl)-butan-2-ol	0.85	0.94	0.1
1-(6'-Methoxy-2'-naphthyl)-but-1-en-3-one (nabumetone related compound A)	0.93	0.25	0.1
Nabumetone	1.0	—	—
5-(6'-Methoxy-2'-naphthyl)-3-methylcyclohex-2-en-1-one	1.2	0.42	0.1
5-(6'-Methoxy-2'-naphthyl)-3-methylcyclohexan-1-one	1.9	1.02	0.1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (w/w, %)
1,5-Di-(6'-methoxy-2'-naphthyl)-pentan-3-one	2.6	0.91	0.1
6,6-Dimethoxy-2,2'-binaphthyl	2.7	0.10	0.3
Any individual unknown impurity	—	—	0.1
Total impurities	—	—	0.8

SPECIFIC TESTS

• **Water Determination,** *Method Ic*⟨921⟩

Sample: 1 g

Acceptance criteria: NMT 0.2%

ADDITIONAL REQUIREMENTS

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

• **USP Reference Standards** ⟨11⟩

USP Nabumetone RS

USP Nabumetone Related Compound A RS

1-(6'-Methoxy-2'-naphthyl)-but-1-en-3-one.

$C_{15}H_{14}O_2$ 226.27

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

Topic/Question	Contact	Expert Committee
NABUMETONE	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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