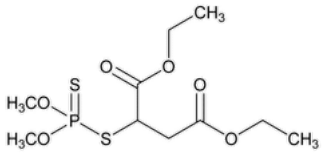
Status: Currently Official on 15-Feb-2025
Official Date: Official as of 01-May-2022
Document Type: USP Monographs
DocId: GUID-3CA17C15-ACBC-48C6-BB81-70D769D9F7A3_3_en-US
DOI: https://doi.org/10.31003/USPNF_M47105_03_01
DOI Ref: j5mf6

Malathion

Change to read:



$C_{10}H_{19}O_6PS_2$ ▲330.35▲ (USP 1-May-2022)

Butanedioic acid, [(dimethoxyphosphinothioyl)-thio]-, diethyl ester, (±)-;

Diethyl (±)-mercaptosuccinate, *S*-ester with *O*,*O*-dimethyl phosphorodithioate;

▲Diethyl 2-[(dimethoxyphosphorothioyl)thio]succinate▲ (USP 1-May-2022) CAS RN®: 121-75-5; UNII: U5N7SU872W.

DEFINITION

Malathion contains NLT 98.0% and NMT 102.0% of malathion ($C_{10}H_{19}O_6PS_2$).

IDENTIFICATION

• **A. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197F

Add the following:

▲• **B.** The retention time of the malathion peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-May-2022)

ASSAY

Change to read:

• **Procedure**

▲**Solution A:** Water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	55	45
20	55	45
25	5	95
30	5	95
30.1	55	45
35	55	45

Diluent: Acetonitrile and water (75:25)

Standard solution: 2 mg/mL of USP Malathion RS in *Diluent*

Sample solution: 2 mg/mL of Malathion in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 210 nm

Column: 4.6-mm × 15-cm; 5-µm packing L1

Column temperature: 35°

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of malathion ($C_{10}H_{19}O_6PS_2$) in the portion of Malathion taken:

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

r_U = peak response of malathion from the *Sample solution*

r_S = peak response of malathion from the *Standard solution*

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

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

▲ (USP 1-May-2022)

Acceptance criteria: 98.0%–102.0%

IMPURITIES

Delete the following:

▲• **Limit of Isomalathion**

Mobile phase: Methanol and water (50:30)

Standard solution: 0.1 mg/mL of USP Isomalathion RS in methanol

Sample solution: 20 mg/mL of Malathion in methanol

Chromatographic system

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

Mode: LC

Detector: UV 210 nm

Column: 4-mm × 30-cm; 10-µm packing L1

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for isomalathion and malathion are about 0.5 and 1.0, respectively.]

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of isomalathion in the portion of Malathion taken:

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

r_U = peak response of isomalathion from the *Sample solution*

r_S = peak response of isomalathion from the *Standard solution*

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

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

Acceptance criteria: NMT 0.3%▲ (USP 1-May-2022)

Add the following:

▲• **Organic Impurities**

Solution A: Acetonitrile and water (43:57)

Solution B: Acetonitrile

Mobile phase: See *Table 2*.

Table 2

Time (min)	Solution A (%)	Solution B (%)
0	100	0
30	100	0
35	10	90
40	10	90
40.1	100	0
48	100	0

Sensitivity solution: 0.01 mg/mL of USP Malathion RS in methanol

Standard solution: 0.02 mg/mL each of USP Malathion RS and malathion impurity B, and 0.06 mg/mL of USP Isomalathion RS in methanol

Sample solution: 20 mg/mL of Malathion in methanol

Chromatographic system

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

Mode: LC

Detector: UV 210 nm

Column: 4.6-mm × 15-cm; 5-µm packing L1

Column temperature: 35°

Flow rate: 1 mL/min

Injection volume: 10 µL

System suitability

Samples: *Sensitivity solution* and *Standard solution*

Suitability requirements

Resolution: NLT 2.0 between malathion impurity B and isomalathion, *Standard solution*

Relative standard deviation: NMT 5.0% each for malathion, isomalathion, and malathion impurity B, *Standard solution*

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of isomalathion in the portion of Malathion taken:

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

r_U = peak response of isomalathion from the *Sample solution*

r_S = peak response of isomalathion from the *Standard solution*

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

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

Calculate the percentage of malathion impurity B in the portion of Malathion taken:

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

r_U = peak response of malathion impurity B from the *Sample solution*

r_S = peak response of malathion impurity B from the *Standard solution*

C_S = concentration of malathion impurity B in the *Standard solution* (mg/mL)

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

Calculate the percentage of any individual impurity in the portion of Malathion taken:

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

r_U = peak response of any individual impurity from the *Sample solution*

r_S = peak response of malathion from the *Standard solution*

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

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

Acceptance criteria: See *Table 3*. The reporting threshold is 0.05%.

Table 3

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Malathion impurity B[a]	0.17	0.10
Isomalathion	0.24	0.3
Malathion	1.0	—
Any individual unspecified impurity	—	0.10
Total impurities[b]	—	1.0

[a] Diethyl 2-[(dimethoxyphosphoryl)thio]succinate.

[b] Total impurities excludes isomalathion.

▲ (USP 1-May-2022)

SPECIFIC TESTS

- **Specific Gravity ⟨841⟩:** Between 1.220 and 1.240
- **Water Determination ⟨921⟩, *Method I*:** NMT 0.1%

ADDITIONAL REQUIREMENTS

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

Change to read:

- **USP Reference Standards ⟨11⟩**

USP Isomalathion RS

▲Diethyl 2-{[methoxy(methylthio)phosphoryl]thio}succinate.▲ (USP 1-May-2022)

$C_{10}H_{19}O_6PS_2$ ▲330.35▲ (USP 1-May-2022)

USP Malathion RS

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

Topic/Question	Contact	Expert Committee
MALATHION	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. 46(5)

Current DocID: GUID-3CA17C15-ACBC-48C6-BB81-70D769D9F7A3_3_en-US

DOI: https://doi.org/10.31003/USPNF_M47105_03_01

DOI ref: j5mf6