Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-Aug-2024
Document Type: NF Monographs
DocId: GUID-6F7D889B-7149-4016-8542-996EFEE563BC_3_en-US
DOI: https://doi.org/10.31003/USPNF_M3062_03_01
DOI Ref: 6xaze

Polyvinyl Acetate Dispersion

DEFINITION

Dispersion of polyvinyl acetate in water. It contains 25.0% to 30.0% of polyvinyl acetate. It may contain povidone and sodium lauryl sulfate as stabilizers.

IDENTIFICATION

• **A. Film Formation:** Place 1 drop of Dispersion on a glass plate and allow to dry. A clear and homogeneous film is formed.

• **B. Infrared Absorption**

(See *Spectroscopic Identification Tests ⟨197⟩, Infrared Spectroscopy*.)

Analysis: Place 1 drop of the Dispersion on a glass plate, and cover the test substance with a water-resistant crystal disk (silver chloride or KRS-5).[1] Gently press on, and then remove the crystal disk. Dry the crystal disk in a drying chamber until a homogeneous film is formed.

Acceptance criteria: The IR absorption spectrum of the film so formed exhibits maxima corresponding to the same wavelengths as those of a similar preparation of USP Polyvinyl Acetate Dispersion RS treated in the same manner.

ASSAY

Change to read:

• **Procedure**

Sample 1: 10 g of Dispersion

Solvent: 50 mL of a mixture of equal volumes of alcohol and petroleum ether with a 100°–120° boiling range, which is previously neutralized with 0.1 N potassium hydroxide or 0.1 N sodium hydroxide

Analysis 1: Dissolve *Sample 1* in the *Solvent*. Add 0.5 mL of phenolphthalein TS, and titrate with 0.1 N potassium hydroxide or 0.1 N sodium hydroxide until the pink color persists for at least 15 s.

Calculate the acid value, I_A:

$$\text{Result} = (M_{r1} \times V \times N)/W$$

M_{r1} = molecular weight of potassium hydroxide, 56.11

V = volume of 0.1 N potassium hydroxide or 0.1 N sodium hydroxide consumed in the actual test (mL)

N = exact normality of the potassium hydroxide solution or sodium hydroxide solution

W = weight of Dispersion taken for the test (g)

Sample 2: 1.5 g of Dispersion

Analysis 2: Transfer *Sample 2* to a 250-mL borosilicate glass flask fitted with a reflux condenser. Add 25.0 mL of 0.5 M alcoholic potassium hydroxide and a few glass beads. Attach the condenser and heat under reflux for 30 min. Add 1 mL of phenolphthalein TS, and titrate immediately (while still hot) with 0.5 N hydrochloric acid VS. Perform a blank determination under the same conditions (see ▲*Titrimetry ⟨541⟩*▲ (CN 1-Aug-2024).)

Calculate the saponification value, I_S:

$$\text{Result} = [M_{r1} \times (V_B - V_T) \times N]/W$$

M_{r1} = molecular weight of potassium hydroxide, 56.11

V_B = volume of 0.5 N hydrochloric acid consumed in the blank test (mL)

V_T = volume of 0.5 N hydrochloric acid consumed in the actual test (mL)

N = exact normality of the hydrochloric acid

W = weight of Dispersion taken for the test (g)

Calculate the percentage content of polyvinyl acetate in the portion of Dispersion taken:

$$\text{Result} = F \times \{M_{r2} \times [(I_S - I_A)/M_{r1}]\} \times 100$$

F = factor converting mg to g, 10^{-3} g/mg

M_{r2} = molecular weight of vinyl acetate, 86.09

I_S = saponification value

I_A = acid value

M_{r1} = molecular weight of potassium hydroxide, 56.11

Acceptance criteria: The content of polyvinyl acetate is 25.0%–30.0%.

OTHER COMPONENTS

Stabilizers

- **Povidone**

[Note—Perform this test only if the Dispersion contains povidone.]

Sample: 0.25 g

Analysis: Perform nitrogen determination by sulfuric acid digestion on the *Sample* as directed in *Nitrogen Determination ⟨461⟩, Method II*.

Calculate the percentage content of povidone in the portion of Dispersion taken:

$$\text{Result} = N/N_V$$

N = percentage content of nitrogen

N_V = percentage content, expressed as a decimal number, of nitrogen in vinylpyrrolidone, 0.126

Acceptance criteria: The content of povidone is NMT 4.0%.

IMPURITIES

- **Residue on Ignition ⟨281⟩**

Sample: 1.0 g of Dispersion

Analysis: Heat a silica crucible to redness for 30 min, allow to cool in a desiccator, and weigh. Evenly distribute the *Sample* in the crucible and weigh. Dry the crucible at 100°–105° for 1 h and ignite in a muffle furnace at 600 ± 25°, until the test substance is thoroughly charred. Continue the experiment as directed in *Residue on Ignition ⟨281⟩* on the residue obtained, beginning with "Moisten the sample with a small amount (usually 1 mL) of sulfuric acid…"

Acceptance criteria: NMT 0.5%

- **Limit of Vinyl Acetate**

Solution A: Acetonitrile, methanol, and water (5:5:90)

Solution B: Acetonitrile, methanol, and water (45:5:50)

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
2	100	0
40	85	15

Time (min)	Solution A (%)	Solution B (%)
42	0	100
48	0	100
51	100	0

Standard solution: Transfer 50 mg of vinyl acetate to a 100-mL volumetric flask, dissolve in and dilute with methanol to volume, and mix well. Dilute 5.0 mL of the solution with *Solution A* to 100 mL. Dilute 10.0 mL of this solution with *Solution A* to 100 mL. The *Standard solution* contains about 2.5 µg/mL of vinyl acetate. [Note—This solution should be analyzed within 1 h when stored at room temperature.]

System suitability solution: Transfer 50 mg of vinyl acetate and 50 mg of 1-vinylpyrrolidin-2-one to a 50-mL volumetric flask, add 10 mL of methanol, sonicate or gently shake the flask to dissolve the materials. Dilute with *Solution A* to volume. Dilute 10 mL of this solution with *Solution A* to 100 mL. Dilute 5 mL of this solution with *Solution A* to 100 mL. The *System suitability solution* contains about 5 µg/mL each of vinyl acetate and 1-vinylpyrrolidin-2-one.

Sample solution: Transfer 250 mg of Dispersion to a 10-mL volumetric flask, add about 4 mL of methanol, and sonicate. After cooling to ambient temperature, dilute with water to volume, and mix. Centrifuge at 4000 × g for 10 min, and pass through a 0.2-µm membrane filter. [Note—This solution should be analyzed within 1 h when stored at room temperature.]

Chromatographic system

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

Mode: LC

Detector: UV 205 nm

Columns

Precolumn: 4.0-mm × 3-cm; 5-µm packing L1 may be used if a matrix effect is observed. [Note—The matrix effect may result in poor reproducibility of the retention times and of the peak shapes.]

Analytical: 4.0-mm × 25-cm; 5-µm packing L1

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 10 µL

System suitability

Sample: *System suitability solution*

[Note—The relative retention times for vinyl acetate and 1-vinylpyrrolidin-2-one are 1.0 and 1.2, respectively.]

Suitability requirements

Resolution: NLT 5.0 between vinyl acetate and 1-vinylpyrrolidin-2-one

Relative standard deviation: NMT 5.0% determined from the 1-vinylpyrrolidin-2-one peak

Analysis

Samples: *Standard solution* and *Sample solution*

Acceptance criteria: The response of the vinyl acetate peak from the *Sample solution* is NMT that of the vinyl acetate peak from the *Standard solution*, corresponding to NMT 100 ppm of vinyl acetate.

- **Limit of Acetic Acid/Acetate**

Solution A: 5 mM sulfuric acid

Solution B: Acetonitrile and 5 mM sulfuric acid (1:1)

Mobile phase: See *Table 2*.

Table 2

Time (min)	Solution A (%)	Solution B (%)
0	100	0
10	100	0
10.5	0	100

Time (min)	Solution A (%)	Solution B (%)
20	0	100
20.5	100	0
30	100	0

System suitability solution: Transfer 30 mg of glacial acetic acid and 30 mg of malonic acid to a 25-mL volumetric flask, dilute with methanol to volume, and mix well. Transfer 1 mL of the solution to a 25-mL flask, dilute with water to volume, and mix well. The solution contains 0.048 mg/mL each of acetic acid and malonic acid.

Standard solution: 0.1 mg/mL of acetic acid in water

Sample solution: Transfer 330 mg of Dispersion to a 50-mL volumetric flask, add about 5 mL of methanol, and dilute with water to volume, which leads to a precipitation of sample. Pass the dispersion through a 0.2-µm regenerated cellulose membrane filter.[2] Use the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 205 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Column temperature: 25°

Flow rate: 1 mL/min

Injection volume: 20 µL

Run time: 30 min

System suitability

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

[NOTE—The relative retention times for malonic acid and acetic acid are 0.9 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between malonic acid and acetic acid, *System suitability solution*

Relative standard deviation: NMT 5.0% determined from the acetic acid peak, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of acetic acid in the portion of Dispersion taken:

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

r_U = peak response of acetic acid from the *Sample solution*

r_S = peak response of acetic acid from the *Standard solution*

C_S = concentration of acetic acid in the *Standard solution* (mg/mL)

C_U = concentration of Polyvinyl Acetate Dispersion in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 1.5% of acetic acid

SPECIFIC TESTS

• **MICROBIAL ENUMERATION TESTS ⟨61⟩** and **TESTS FOR SPECIFIED MICROORGANISMS ⟨62⟩:** The total aerobic microbial count is NMT 1000 cfu/g, and the total combined molds and yeasts count is NMT 100 cfu/g.

• **pH ⟨791⟩:** 3.0–5.5

• **LOSS ON DRYING ⟨731⟩**

Sample: 1.0 g of Dispersion

Analysis: Dry the *Sample* at 110° for 5 h.

Acceptance criteria: 68.5%–71.5%

• **COAGULUM CONTENT**

Sample: 100 g of Dispersion

Analysis: Accurately weigh a stainless steel sieve with 45-µm openings or a suitable single-woven wire cloth with a mesh width of 45 µm, and filter the *Sample* through it. [NOTE—Suitable single-woven wire cloth mesh meets the requirements set in ISO 9044.] Wash the sieve or the cloth with distilled water until a clear filtrate is obtained, and dry the sieve or the cloth to constant weight at 100°–105°.

Acceptance criteria: The weight of the residue is NMT 500 mg (0.5%).

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers at a temperature below 25°. Protect from freezing.
- **LABELING:** Label it to indicate the amounts of povidone and sodium lauryl sulfate.
- **USP REFERENCE STANDARDS** ⟨11⟩
  USP Polyvinyl Acetate Dispersion RS

[1] KRS-5 consists of 42% thallium(I) bromide and 58% thallium(I) iodine by molecular weight. Suitable disks of silver chloride and of KRS-5 are available from www.crystals.saint-gobain.com, www.almazoptics.com, and www.internationalcrystal.net.

[2] Whatman Spartan HPLC certified syringe filter, Whatman Cat # 10463060 or equivalent filter.

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

Topic/Question	Contact	Expert Committee
POLYVINYL ACETATE DISPERSION	Documentary Standards Support	CE2020 Complex Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 41(5)

Current DocID: GUID-6F7D889B-7149-4016-8542-996EFEE563BC_3_en-US
DOI: https://doi.org/10.31003/USPNF_M3062_03_01
DOI ref: 6xaze