Status: Currently Official on 13-Feb-2025
Official Date: Official as of 01-Jun-2023
Document Type: NF Monographs
DocId: GUID-7D055B95-B5F9-4F5A-9F11-DE521D877BF4_4_en-US
DOI: https://doi.org/10.31003/USPNF_M7683_04_01
DOI Ref: a7fm9

Purified Bentonite

DEFINITION

Purified Bentonite is a colloidal montmorillonite that has been processed to remove grit and nonswellable ore components.

IDENTIFICATION

• **A. X-Ray Powder Diffraction ⟨941⟩**

Sample: 2 g

Analysis 1: Add the *Sample* in small portions to 100 mL of water with intense agitation. Allow to stand for 12 h to ensure complete hydration. Place 2 mL of the resulting mixture on a suitable glass slide, and allow to air-dry at room temperature to produce an oriented film. Place the slide in a vacuum desiccator over a free surface of ethylene glycol. Evacuate the desiccator, and close the stopcock so that the ethylene glycol saturates the desiccator chamber. Allow to stand for 12 h. Record the X-ray diffraction pattern, and calculate the *d* values.

Acceptance criteria 1: The largest peak corresponds to a *d* value between 15.0 and 17.2 Å.

Analysis 2: Prepare a random powder specimen of Purified Bentonite, record the X-ray diffraction pattern, and determine the *d* values in the region between 1.48 and 1.54 Å.

Acceptance criteria 2: A peak is found at 1.492–1.504 Å.

IMPURITIES

Change to read:

• ▲**Arsenic ⟨211⟩, *Procedures, Procedure 1*▲** (CN 1-Jun-2023)

Standard solution: 5 mL of *Standard Arsenic Solution* (5 µg of As)

Sample solution: Transfer 13.3 g to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), mix, cover with a watch glass, and boil gently, with occasional stirring, for 15 min without allowing excessive foaming. Allow the insoluble material to settle, and decant the hot supernatant through a rapid-flow filter paper into a 200-mL volumetric flask, retaining as much sediment as possible in the beaker. Add 25 mL of hot dilute hydrochloric acid (1 in 25) to the residue in the beaker, stir, heat to boiling, allow the insoluble material to settle, and decant the supernatant through the filter into the 200-mL volumetric flask. Repeat the extraction with four additional 25-mL portions of hot dilute hydrochloric acid (1 in 25), decanting each hot supernatant through the filter into the volumetric flask. At the last extraction, transfer as much of the insoluble material as possible onto the filter. Cool the combined filtrates to room temperature, and add dilute hydrochloric acid (1 in 25) to volume.

Analysis: Using a 25-mL aliquot of *Sample solution* and the *Standard solution*, treat them as directed in the *Procedure*.

Acceptance criteria: NMT 3 µg/g; the absorbance due to any red color from the *Sample solution* does not exceed that produced by the *Standard solution*.

• **Lead**

[Note—The *Standard solution* and the *Sample solution* may be modified, if necessary, to obtain solutions of suitable concentrations, adaptable to the linear or working range of the instrument.]

Standard solution: On the day of use, dilute 3.0 mL of lead nitrate stock solution TS to 100 mL. Each milliliter of the *Standard solution* contains the equivalent of 3 µg of lead.

Sample solution: Transfer 10.0 g of Purified Bentonite to a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), stir, cover with a watch glass, and boil for 15 min. Cool to room temperature, and allow the insoluble matter to settle. Decant the supernatant through a rapid-flow filter paper into a 400-mL beaker. Add 25 mL of hot water to the insoluble matter in the 250-mL beaker, stir, allow the insoluble matter to settle, and decant the supernatant through the filter into the 400-mL beaker. Repeat the extraction with two additional 25-mL portions of water, decanting each supernatant portion through the filter into the 400-mL beaker. Wash the filter with 25 mL of hot water, collecting this filtrate in the 400-mL beaker. Concentrate the combined extracts by gentle boiling to approximately 20 mL. If a precipitate appears, add 2–3 drops of nitric acid, heat to boiling, and cool to room temperature. Pass the concentrated extracts through a rapid-flow filter paper into a 50-mL volumetric flask. Transfer the remaining contents of the 400-mL beaker through the filter paper and into the flask with water. Dilute with water to volume.

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Atomic absorption spectrophotometer equipped with a deuterium arc background correction and a single-slot burner

Analytical wavelength: 284 nm

Lamp: Lead hollow-cathode

Flame: Air–acetylene

Analysis: Determine the absorbances of the *Standard solution* and the *Sample solution*.

Acceptance criteria: The absorbance of the *Sample solution* is NMT that of the *Standard solution* (15 µg/g).

SPECIFIC TESTS

• **Microbial Enumeration Tests ⟨61⟩** and **Tests for Specified Microorganisms ⟨62⟩:** The total aerobic microbial count does not exceed 1×10^3 cfu/g. It meets the requirements of the test for absence of *Escherichia coli*.

• **pH ⟨791⟩**

Sample suspension: 50 mg/mL

Acceptance criteria: 9.0–10.0

• **Loss on Drying ⟨731⟩**

Analysis: Dry at 110° to constant weight.

Acceptance criteria: NMT 8.0%

• **Viscosity**

Sample: After determining *Loss on Drying*, weigh a quantity of Purified Bentonite equivalent to 25.0 g on the dried basis. Over a period of a few seconds, transfer the undried sample specimen to a suitable 1-L blender jar containing an amount of water, maintained at a temperature of 25 ± 2°, that is sufficient to produce a mixture weighing 500 g. Blend for 3 min, accurately timed, at 14,000–15,000 rpm (high speed). [1]

[Note—Heat generated during blending causes a temperature rise to 33 ± 3°.]

Analysis: Transfer the contents of the blender to a 600-mL beaker and allow to stand for 5 min. Using a suitable rotational viscometer[2] equipped with a spindle having a cylinder 1.87 cm in diameter and 0.69 cm high attached to a shaft 0.32 cm in diameter, the distance from the top of the cylinder to the lower tip of the shaft being 2.54 cm, and the immersion depth being 5.00 cm (No. 2 spindle), operate the viscometer at 60 rpm for 6 min, accurately timed, and record the scale reading.

Acceptance criteria: 40–200 mPa · s

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

[1] A suitable blender is available from Waring as Waring Commercial Blender Model 7009G or equivalent with 1-L glass jar and tachometer adapter, Model CAC24 or equivalent.

[2] A suitable viscometer is available from Brookfield as Viscometer Model LVF or LVT, or equivalent.

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 44(5)

Current DocID: GUID-7D055B95-B5F9-4F5A-9F11-DE521D877BF4_4_en-US

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