

Ferrocyanides Limit Test

Ferrocyanides Limit Test — Summary

Purpose: The ferrocyanides limit test for Sodium Chloride checks that **ferrocyanide/ferrocyanide-derived cyanide species** are not present at levels that produce a visible blue complex when reacted with iron(III). **Acceptance:** the test solution must show **no blue color within 10 minutes** after addition of the reagents.

Principle (brief)

Dissolved sample is treated with a mixture of **ferric (Fe^{3+})** and **ferrous (Fe^{2+})** reagents; if ferrocyanide is present, it reacts with ferric ions to form **Prussian blue** (a deep blue/blue-green precipitate or color). The absence of blue color within the specified observation time indicates compliance.

Step-by-step procedure (bench-ready, follow official monograph/SOP exactly)

“ **Note:** The steps below follow the USP monograph wording and practical bench practice. For official QC work, use the exact monograph text and your validated SOP.

- 1. Reagents and solutions (prepare fresh)**
 - **Ferric ammonium sulfate solution:** 1 g ferric ammonium sulfate per 100 mL of 0.1 N sulfuric acid (use this as prepared in the monograph).
 - **Ferrous sulfate solution:** 1 in 100 (i.e., dilute stock as specified by your SOP).
 - **Purified (iodide/contaminant-free) water.**
- 2. Weigh and dissolve sample**
 - Accurately weigh **2.0 g** of the sodium chloride sample.
 - Dissolve the 2.0 g in **6 mL** of purified water in a clean test tube or small beaker. Mix until fully dissolved.
- 3. Prepare the iron reagent mixture**

- Prepare a mixture consisting of **5 mL** of the ferric ammonium sulfate solution and **95 mL** of the ferrous sulfate solution; then take **0.5 mL** of this mixture for the test. (Prepare the larger mixture fresh and use the 0.5 mL aliquot immediately.)
- 4. Add reagent to sample**
 - To the dissolved sample (2.0 g in 6 mL water), add **0.5 mL** of the iron reagent mixture. Mix gently to combine.
 - 5. Incubate and observe**
 - Allow the mixture to stand **for 10 minutes** at ambient temperature.
 - **Read the result:** examine the solution in natural light against a neutral background. **No blue color** developing within 10 minutes = **pass**; any blue color = **fail**.
 - 6. Controls**
 - Run a **blank** (6 mL water + 0.5 mL reagent) to confirm reagent background.
 - Optionally run a **positive control** spiked near the limit to confirm the operator can detect a faint blue.

Practical tips for reliable performance

- **Fresh reagents:** Prepare the ferric/ferrous reagent mixture immediately before use; ferrous solutions oxidize on standing and reagent ratios affect sensitivity.
- **Clean glassware:** Use clean, unscratched test tubes and rinse with purified water to avoid false color or adsorption artifacts.
- **Consistent lighting and background:** Read against a neutral (white or gray matte) background under consistent ambient light to detect faint blue hues.
- **Mixing:** Add the reagent gently and invert or swirl; avoid vigorous agitation that can entrain air or cause splashing.
- **Timing:** Start a timer immediately after reagent addition and read at the specified 10-minute mark; do not rely on later or earlier observations.

Cautions and safety

- **Chemical handling:** Ferric and ferrous salts and the acidic ferric ammonium sulfate solution can be **irritants**; wear safety goggles, gloves, and a lab coat. Prepare and handle reagents in a well-ventilated area.
- **Oxidation and stability:** Ferrous sulfate oxidizes to ferric species on exposure to air; store and prepare under conditions recommended by your SOP and discard aged solutions.
- **Waste disposal:** Solutions containing iron and any cyanide/ferrocyanide reaction products must be collected and disposed of as hazardous chemical waste per institutional and regulatory procedures—do not pour to drain.

- **Contamination control:** Avoid cross-contamination from glassware or pipettes previously used with cyanide/ferrocyanide or iron reagents.

Quick bench checklist

- **Reagents prepared:** ferric ammonium sulfate solution; ferrous sulfate solution; purified water.
 - **Sample weighed:** 2.0 g dissolved in 6 mL water.
 - **Reagent aliquot ready:** 0.5 mL of the iron reagent mixture.
 - **Timer set:** observe at 10 minutes.
 - **Blank and positive control:** prepared and run concurrently.
 - **PPE on:** goggles, gloves, lab coat; waste container labeled.
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