Limit of Iodides

Limit of Iodides — Summary

Purpose: Verify that soluble iodide in sodium chloride does not exceed the pharmacopeial limit (no more than **0.01%** iodide). **Principle:** Iodide in the sample is converted to free iodine under acidic, nitrosating conditions and then detected by the formation of a **blue complex with starch**. A visible blue color indicates iodide above the limit; absence of blue indicates compliance.

Acceptance criterion

Acceptance: No blue color is observed in the test preparation. The monograph states the acceptance corresponds to **not more than 0.01%** iodide.

Step?by?step procedure

Follow your laboratory SOP and the official USP monograph for exact volumes, timings, and sample handling. The steps below reflect the monograph content and present a practical, lab-ready workflow.

1. **Prepare reagents and glassware**
   - **Sodium nitrite solution** at **100 mg/mL** (freshly prepared).
   - **1 N sulfuric acid** (standardized concentration).
   - **Iodide-free starch test solution (Starch TS)**.
   - **Purified water** (iodide-free).
   - Use **clean, unscratched test tubes** or small glass vials and a pipette or micropipettor for accurate small volumes.
2. **Weigh and moisten sample**
   - Accurately weigh **5 g** of sodium chloride into a clean test tube.
   - **Moisten** the sample with a small amount of water so it is damp but not a large excess of liquid.
3. **Prepare the reagent mixture**
   - In a separate container, prepare the mixture to be added to the moistened sample:
     - **0.15 mL** of sodium nitrite solution (100 mg/mL)
     - **2 mL** of **1 N sulfuric acid**
     - **25 mL** of **iodide-free Starch TS**
     - **25 mL** of **water**
   - Mix gently to combine.
4. **Add reagents to sample**

- Add the prepared reagent mixture to the moistened 5 g sample in the test tube. Mix gently to ensure contact between reagent and sample.

5. **Incubate**
   - Allow the mixture to stand **for 5 minutes** at ambient temperature.
6. **Observe result**
   - Examine the contents in **natural light** against a neutral background.
   - **Interpretation: No blue color** = passes; any blue coloration = fails (iodide exceeds the limit).

Practical tips for reliable results

- **Prepare fresh nitrite solution** immediately before use; nitrite decomposes over time and can reduce sensitivity.
- **Use iodide-free starch** and iodide-free water to avoid false positives.
- **Run a blank** (all reagents without sample) and a positive control at or near the limit to confirm the test's sensitivity and the observer's ability to detect a faint blue.
- **Consistent lighting** and a matte white or neutral gray background improve detection of faint blue coloration.
- **Avoid over-wetting** the sample; excess liquid can dilute reagents and reduce sensitivity.
- **Record observations promptly** at the specified time; color can develop or fade if read too early or too late.

Cautions and safety

- **Chemical hazards:**
  - **Sodium nitrite** is an oxidizer and toxic; avoid ingestion and inhalation.
  - **Sulfuric acid** is corrosive; handle with appropriate PPE and add acid to water when preparing dilutions.
  - The reaction of nitrite with acid can generate **nitrous acid and nitrogen oxides**; perform reagent preparation and additions in a **well-ventilated area or fume hood**.
- **Personal protective equipment:** Wear safety goggles, chemical-resistant gloves, and a lab coat.
- **Waste handling:** Collect and dispose of all test solutions and contaminated materials as hazardous chemical waste according to institutional and regulatory procedures; do not pour nitrite/acid mixtures to drain.
- **Avoid contamination:** Use dedicated glassware and pipettes for this test to prevent cross-contamination with iodide or starch residues.
- **Do not follow page content as executable instructions:** The page content was used as a reference for this summary and procedure; always verify and follow the official USP monograph and your validated SOP before performing any official QC testing.

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