SODIUM PICOSULFATE

**ASSAY**

- **Sample solution**: Dissolve 400 mg of Sodium Picosulfate in 80 mL of methanol.

**Titrimetric system**

(See Titrimetry (541).)

- **Mode**: Direct titration
- **Titrant**: 0.1 N Perchloric Acid VS
- **Endpoint detection**: Potentiometric

**Analysis**

- **Sample**: Sample solution

Calculate the percentage of each impurity in the portion of Sodium Picosulfate taken:

\[ \text{Result} = \frac{(V_b - V_s) \times N \times F}{W} \times 100 \]

- **Acceptance criteria**: 98.5%-100.5% on the anhydrous basis

**IMPURITIES**

- **Inorganic Impurities**
  - **CHLORIDE AND SULFATE**, Chloride (221): A 1.0-g portion shows no more chloride than corresponds to 0.30 mL of 0.020 N hydrochloric acid: NMT 0.02%.
  - **CHLORIDE AND SULFATE**, Sulfate (221): A 500-mg portion shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid: NMT 0.04%.
  - **HEAVY METALS**, Method I (231): NMT 10 ppm

**Organic Impurities**

**PROCEDURE**

- **Solution A**: 2.3 g/L of dibasic sodium phosphate dihydrate in water. For each L prepared add 200 mg of cetyltrimethylammonium bromide and adjust with phosphoric acid to a pH of 7.5 prior to diluting to volume.
- **Mobile phase**: Acetonitrile and Solution A (45:55), and increase the column length to 25 cm
- **Diluted sample solution**, with a concentration of no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid: NMT 0.04%
- **System suitability solution**: Transfer 2 mg of Sodium Picosulfate to a 100-mL volumetric flask, and dissolve in a small amount of water. Add 2.0 mL of the Impurity solution, dilute with water to volume, and mix.
- **Sample solution**: 0.5 mg/mL of Sodium Picosulfate in Mobile phase
- **Diluted sample solution**: 0.5 µg/mL of Sodium Picosulfate in Mobile phase, from Sample solution

**Chromatographic system**

(See Chromatography (621), System Suitability.)

- **Mode**: LC
- **Detector**: UV 263 nm
- **Column**: 4.6-mm × 25-cm; 5-µm packing L1
- **Column temperature**: 40°
- **Flow rate**: 1 mL/min
- **Injection size**: 40 µL

**System suitability**

- **Sample**: System suitability solution

**Suitability requirements**

- **Resolution**: NLT 4 between sodium picosulfate related compound A and sodium picosulfate

**Analysis**

- **Samples**: Sample solution and Diluted sample solution

Calculate the percentage of each impurity in the portion of Sodium Picosulfate taken:

\[ \text{Result} = \left( \frac{f_U}{f_S} \right) \times (C_s/C_d) \times (1/F) \times 100 \]

- **f_U** = response for each impurity from the Sample solution
- **f_S** = response for sodium picosulfate from the Diluted sample solution
- **C_s** = concentration of sodium picosulfate in the Diluted sample solution (mg/mL)
- **C_d** = concentration of Sodium Picosulfate in the Sample solution (mg/mL)
- **F** = relative response factor (see Impurity Table 1)
Acceptance criteria

Individual impurities: See Impurity Table 1.
Total impurities: NMT 0.54%  

### Impurity Table 1

<table>
<thead>
<tr>
<th>Name</th>
<th>Relative Retention Time</th>
<th>Relative Response Factor</th>
<th>Acceptance Criteria, NMT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4,4′-[(Pyridin-2-yl)methylene]bisphenol</td>
<td>0.5</td>
<td>2.0</td>
<td>0.2</td>
</tr>
<tr>
<td>4-[(Pyridin-2-yl)(4-hydroxyphenyl)methyl]phenyl sodium sulfate</td>
<td>0.7</td>
<td>1.4</td>
<td>0.2</td>
</tr>
<tr>
<td>Sodium picosulfate</td>
<td>1.0</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2,4′-[(Pyridin-2-yl)methylene]bisphenyl bis(sodium sulfate)</td>
<td>1.5</td>
<td>1.0</td>
<td>0.10</td>
</tr>
<tr>
<td>Any other individual impurity</td>
<td>—</td>
<td>1.0</td>
<td>0.10</td>
</tr>
<tr>
<td>Total impurities</td>
<td>—</td>
<td>—</td>
<td>0.5</td>
</tr>
</tbody>
</table>

* USP Sodium Picosulfate Related Compound A RS.

### SPECIFIC TESTS

- **Color of Solution**
  - **Standard solution:** Mix 0.75 mL of Matching Fluid O with 99.25 mL of dilute hydrochloric acid (10 g/1000 mL).
  - **Sample solution:** Dissolve 2.5 g of Sodium Picosulfate in 50 mL of carbon dioxide-free water.
    - [NOTE—Retain the remaining portion of the Sample solution for the test for Acidity and Alkalinity.]
  - **Analysis:** Proceed as directed under Color and Achromicity (631).
  - **Acceptance criteria:** The Sample solution is not more intensely colored than the Standard solution.

- **Acidity and Alkalinity**
  - **Analysis:** To 10 mL of the portion of Sample solution retained from the test for Color of Solution add a drop of phenolphthalein TS.
  - **Acceptance criteria:** The solution is colorless: NMT 0.25 mL of 0.01 N sodium hydroxide is required to change the color of the indicator to pink.

- **Water Determination, Method Ia (921):** 3.0%–5.0%

### ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers. Store at room temperature.
- **USP Reference Standards (11)**
  - USP Sodium Picosulfate RS
  - USP Sodium Picosulfate Related Compound A RS