Potassium Perchlorate

» Potassium Perchlorate contains not less than 99.0 percent and not more than 100.5 percent of $KClO_4$, calculated on the dried basis.

Caution: Great care should be taken in handling Potassium Perchlorate in solution or in the dry state, as explosions may occur if it is brought into contact with organic or other readily oxidizable substances.

Packaging and storage—Preserve in well-closed containers.

Identification—

A: Ignite a small portion of a solution (1 in 10) on a platinum wire in a nonluminous flame: a transient violet color is imparted to the flame.

B: Add a few drops of methylene blue solution (1 in 1000) to the solution (1 in 10): a violet-colored precipitate is formed.

pH ⟨791⟩: between 5.0 and 6.5, in a 0.1 M solution.

Loss on drying ⟨731⟩**—**Dry it over silica gel for 12 hours: it loses not more than 0.5% of its weight.

Insoluble substances—Dissolve 20 g in 150 mL of warm water, pass through a tared medium-porosity filtering crucible, and wash with three 50-mL portions of warm water. Dry the residue at 105° for 3 hours: the weight of the residue does not exceed 1 mg (0.005%).

Chloride ⟨221⟩**—**A 5.0-g portion shows no more chloride than corresponds to 0.20 mL of 0.020 N hydrochloric acid (0.003%).

Heavy metals, *Method I* ⟨231⟩: 0.001%.

Limit of sodium—Ignite a small portion of a solution (1 in 10) on a platinum wire in a nonluminous flame: no pronounced yellow color is imparted to the flame.

Organic volatile impurities, *Method I* ⟨467⟩**:** meets the requirements.

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Assay—

Mobile phase—Transfer 16.6 g of phthalic acid to a 100-mL volumetric flask, dissolve in and dilute with methanol to volume, and mix. Transfer 10.0 mL of this solution to a 1000-mL flask, dilute with water to volume, and mix. Adjust with about 450 mg of lithium hydroxide to a pH of 4.5, filter, and degas.

Standard preparation—Transfer about 50 mg of potassium perchlorate, accurately weighed, to a 50-mL volumetric flask, dilute with water to volume, and mix. Transfer 10.0 mL of this solution to a 100-mL volumetric flask, dilute with water to volume, and mix to obtain a solution having a known concentration of about 0.1 mg per mL.

Assay preparation—Using about 50 mg of Potassium Perchlorate, accurately weighed, proceed as directed for the *Standard preparation*.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a conductivity detector and a 4.6-mm × 7.5-cm column that contains 6-µm packing L23. The flow rate is about 1.2 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the tailing factor is not more than 1.5; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 50 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $KClO_4$ in the portion of Potassium Perchlorate taken by the formula:

$$500C(r_U / r_S)$$

in which C is the concentration, in mg per mL, of potassium perchlorate in the *Standard preparation;* and r_U and r_S are the peak responses obtained from the *Assay preparation* and the *Standard preparation*, respectively.