Edition: BP 2025 (Ph. Eur. 11.6 update)

Potassium Perchlorate

General Notices

(Ph. Eur. monograph 1987)

$KClO_4$ 138.6 *7778-74-7*

Action and use

Diagnostic aid; treatment of hyperthyroidism.

Ph Eur

DEFINITION

Content

99.0 per cent to 102.0 per cent.

CHARACTERS

Appearance

White or almost white, crystalline powder or colourless crystals.

Solubility

Sparingly soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

A. Dissolve 0.1 g in 5 mL of *water R*. Add 5 mL of *indigo carmine solution R* and heat to boiling. The colour of the solution does not disappear.
B. Chlorates and chlorides (see Tests).
C. Heat 10 mg over a flame for 2 min. Dissolve the residue in 2 mL of *water R*. The solution gives reaction (a) of chlorides (*2.3.1*).
D. Dissolve 50 mg with heating in 5 mL of *water R*. Allow to cool to room temperature. The solution gives reaction (a) of potassium (*2.3.1*).

TESTS

Solution S

Suspend 5.0 g in 90 mL of *distilled water R* and heat to boiling. Allow to cool. Filter. Dilute the filtrate to 100 mL with *carbon dioxide-free water R*.

Appearance of solution

The solution is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

Dissolve 0.20 g in *water R* and dilute to 20 mL with the same solvent.

Acidity or alkalinity

To 5 mL of solution S add 5 mL of *water R* and 0.1 mL of *phenolphthalein solution R*. Not more than 0.25 mL of *0.01 M sodium hydroxide* is required to change the colour of the indicator. To 5 mL of solution S, add 5 mL of *water R* and 0.1 mL of *bromocresol green solution R*. Not more than 0.25 mL of *0.01 M hydrochloric acid* is required to change the colour of the indicator.

Chlorates and chlorides (*2.4.4*)

Maximum 100 ppm (calculated as chlorides).

To 5 mL of solution S, add 5 mL of *water R* and heat to boiling. Add 1 mL of *nitric acid R* and 0.1 g of *sodium nitrite R*. Allow to cool to room temperature. Dilute to 15 mL with *water R*. The solution complies with the limit test for chlorides. Prepare the standard using 5 mL of *chloride standard solution (5 ppm Cl) R* and 10 mL of *water R*, and adding only 1 mL of *dilute nitric acid R*.

Sulfates (*2.4.13*)

Maximum 100 ppm, determined on solution S.

Prepare the standard using a mixture of 7.5 mL of *sulfate standard solution (10 ppm SO_4) R* and 7.5 mL of *water R*.

Calcium (*2.4.3*)

Maximum 100 ppm, determined on solution S.

Prepare the standard using a mixture of 7.5 mL of *calcium standard solution (10 ppm Ca) R*, 1 mL of *dilute acetic acid R* and 7.5 mL of *distilled water R*.

ASSAY

Prepare a chromatography column 0.3 m long and 10 mm in internal diameter and filled with 10 g of *strongly acidic ion-exchange resin R* covered with *carbon dioxide-free water R*. Maintain a 1 cm layer of liquid above the resin throughout the determination. Allow 100 mL of *dilute hydrochloric acid R* to run through the column at a flow rate of about 5 mL/min. Wash the column (with the tap completely open) with *carbon dioxide-free water R* until the eluate is neutral to *blue litmus paper R*. Dissolve 0.100 g of the substance to be examined in 10 mL of *carbon dioxide-free water R* in a beaker and transfer it to the column reservoir, allow the solution to run through the column at a flow rate of about 3 mL/min and collect the eluate. Wash the beaker 3 times with 10 mL of *carbon dioxide-free water R* and transfer this solution at the same flow rate to the column before it runs dry. Finally, wash the column with 200 mL of *carbon dioxide-free water R* (with the tap completely open) until the eluate is neutral to *blue litmus paper R*. Titrate the combined eluate and washings with *0.1 M sodium hydroxide*, using 1 mL of *phenolphthalein solution R* as indicator.

1 mL of *0.1 M sodium hydroxide* is equivalent to 13.86 mg of $KClO_4$.