

quinolin-8-ol,  $C_9H_7NO$ , and not less than 29.5% and not more than 32.5% of  $K_2SO_4$ , calculated with reference to the anhydrous substance.

#### CHARACTERISTICS

A pale yellow, microcrystalline powder; odourless or almost odourless. It partly liquefies between  $172^\circ$  and  $184^\circ$ .

Freely soluble in *water*; insoluble in *ether*. On extraction with hot *absolute ethanol* a residue of potassium sulphate and a solution of quinolin-8-ol sulphate are obtained.

#### IDENTIFICATION

A. To 5 ml of a 5% w/v solution add drop wise, with shaking, 5M *sodium hydroxide* until a heavy precipitate is produced. Filter, wash with *water* and dry at a pressure not exceeding 0.7 kPa for 3 hours. The *infrared absorption spectrum* of the residue, Appendix II A, is concordant with the *reference spectrum* of quinolin-8-ol (RS 310).

B. To 5 ml of a 5% w/v solution add 0.5 ml of *iron(III) chloride solution R1*. A dark green colour is produced.

C. Yields reaction A characteristic of *potassium salts*, Appendix VI.

D. Yields reaction A characteristic of *sulphates*, Appendix VI.

#### Water

Not more than 5.0% w/w, Appendix IX C. Use 0.5 g.

#### ASSAY

##### For quinolin-8-ol

Dissolve 0.35 g in 50 ml of *water* and 20 ml of *hydrochloric acid*, add 50 ml of 0.05M *bromine VS*, stopper the flask and shake for 15 minutes. Allow to stand for 15 minutes, add 80 ml of *water* and 10 ml of *dilute potassium iodide solution* and titrate with 0.1M *sodium thiosulphate VS* using *starch mucilage*, added towards the end of the titration, as indicator. Repeat the operation without the substance being examined. The difference between the titrations represents the amount of bromine required. Each ml of 0.05M *bromine VS* is equivalent to 3.629 mg of  $C_9H_7NO$ .

##### For potassium sulphate

Prepare a solution of suitable concentration with *water*. Carry out the method for *atomic emission spectrophotometry*, Appendix II D, measuring at 766.5 nm and using *potassium standard solution (600 ppm K)*, suitably diluted with *water*, to prepare the standard solutions. Each mg of potassium is equivalent to 2.2284 mg of  $K_2SO_4$ .

#### Action and use

Used in treatment of acne.

#### Preparation

Potassium Hydroxyquinoline Sulphate and Benzoyl Peroxide Cream.

## Potassium Iodate

$KIO_3$

214.0

7758-05-6

#### DEFINITION

Potassium Iodate contains not less than 99.0% and not more than 101.0% of  $KIO_3$ , calculated with reference to the dried substance.

#### CHARACTERISTICS

A white crystalline powder; odour, slight.

Slowly soluble in *water*; insoluble in *ethanol* (96%).

Dissolve 10 g of the substance being examined in sufficient *water* to produce 200 ml (solution S1).

Add 25 ml of *hydrochloric acid* to 6 g of the substance being examined, evaporate to dryness and repeat. Heat until iodine is removed. Dissolve the residue in 2.5 ml of a 25% v/v solution of *hydrochloric acid* and dilute to 50 ml with *water* (solution S2).

#### IDENTIFICATION

A. 1 ml of solution S1 yields reaction B characteristic of *potassium salts*, Appendix VI.

B. Dissolve 0.1 g in 5 ml of *water*. Add 1 ml of *silver nitrate solution* followed by 1 ml of *sulphur dioxide solution*. A yellow precipitate is produced immediately.

#### Acidity or alkalinity

pH of solution S1, 5.0 to 8.0, Appendix V L.

#### Clarity and colour of solution

Solution S1 is *clear*, Appendix IV A, and *colourless*, Appendix IV B, Method II.

#### Chloride, chlorate, bromide, bromate

Dilute 5 ml of solution S1 to 15 ml with *water*, add 20 ml of *sulphur dioxide solution* and heat on a water bath for 30 minutes. Heat to boiling, cool, add 10 ml of 18M *ammonia* and 20 ml of *silver nitrate solution R2* and dilute to 70 ml with *water*. Filter, transfer 35 ml of the filtrate to a *Nessler cylinder* and acidify with 6 ml of *nitric acid*. After 5 minutes, any opalescence, when viewed vertically, is not greater than that produced by treating 5 ml of a 0.00165% w/v solution of *sodium chloride* at the same time and in the same manner (0.02%).

#### Iodide

Add 1 ml of 1.8M *sulphuric acid* to 25 ml of solution S1 and shake with 1 ml of *chloroform*. Any violet colour produced is not more intense than that of a solution prepared at the same time and in the same manner but using 5 ml of solution S1 and 2 ml of *iodide standard solution (10 ppm I)* (20 ppm).

#### Sulphate

Add 1 ml of a 25% w/v solution of *barium chloride* to 1.5 ml of *sulphate standard solution (10 ppm  $SO_4$ )*, shake and allow to stand for 1 minute. Add 12.5 ml of solution S2 diluted to 15 ml with *distilled water* and 0.5 ml of 5M *acetic acid* and allow to stand for 5 minutes. Any opalescence produced is not more intense than that of a standard prepared in the same manner but using 7.5 ml of *sulphate standard solution (10 ppm  $SO_4$ )* diluted to 15 ml with *distilled water* in place of the solution being examined (50 ppm).

#### Heavy metals

Adjust the pH of 20 ml of solution S2 to about 5 with 5M *ammonia*. The solution complies with *limit test A for heavy metals*, Appendix VII. Use 10 ml of *lead standard solution (2 ppm Pb)* to prepare the standard (20 ppm).

#### Loss on drying

When dried at  $130^\circ$  for 1 hour, loses not more than 0.5% of its weight. Use 1 g.

#### ASSAY

To 1.5 g add sufficient *water* to produce 250 ml. To 25 ml of the resulting solution in an iodine flask add 3 g of *potassium iodide*, 100 ml of *water* and 10 ml of *hydrochloric acid*. Close the flask and stand in the dark for 5 minutes. Titrate the solution with 0.1M *sodium thiosulphate VS* to a light straw colour and then complete the titration to a colourless end point using *starch mucilage* as indicator. Each ml of 0.1M *sodium thiosulphate VS* is equivalent to 3.567 mg of  $KIO_3$ .

**Action and use**

Iodine supplement for emergency use.

**Preparation**

Potassium Iodate Tablets

**Potassium Iodide**

(Ph Eur monograph 0186)

KI

166.0

7681-11-0

Ph Eur

**DEFINITION**

Potassium iodide contains not less than 99.0 per cent and not more than the equivalent of 100.5 per cent of KI, calculated with reference to the dried substance.

**CHARACTERS**

A white powder or colourless crystals, very soluble in water, freely soluble in glycerol, soluble in alcohol.

**IDENTIFICATION**

A. Solution S (see Tests) gives the reactions of iodides (2.3.1).

B. Solution S gives the reactions of potassium (2.3.1).

**TESTS****Solution S**

Dissolve 10.0 g in *carbon dioxide-free water R* prepared from *distilled water R* and dilute to 100 ml with the same solvent.

**Appearance of solution**

Solution S is clear (2.2.1) and colourless (2.2.2, *Method II*).

**Alkalinity**

To 12.5 ml of solution S add 0.1 ml of *bromothymol blue solution R1*. Not more than 0.5 ml of 0.01M *hydrochloric acid* is required to change the colour of the indicator.

**Iodates**

To 10 ml of solution S add 0.25 ml of *iodide-free starch solution R* and 0.2 ml of *dilute sulphuric acid R* and allow to stand protected from light for 2 min. No blue colour develops.

**Sulphates (2.4.13)**

10 ml of solution S diluted to 15 ml with *distilled water R* complies with the limit test for sulphates (150 ppm).

**Thiosulphates**

To 10 ml of solution S add 0.1 ml of *starch solution R* and 0.1 ml of 0.005M *iodine*. A blue colour is produced.

**Heavy metals (2.4.8)**

12 ml of solution S complies with limit test A for heavy metals (10 ppm). Prepare the standard using *lead standard solution (1 ppm Pb) R*.

**Iron (2.4.9)**

5 ml of solution S diluted to 10 ml with *water R* complies with the limit test for iron (20 ppm).

**Loss on drying (2.2.32)**

Not more than 1.0 per cent, determined on 1.00 g of previously powdered substance by drying in an oven at 100°C-105°C for 3 h.

**ASSAY**

Dissolve 1.500 g in *water R* and dilute to 100.0 ml with the same solvent. To 20.0 ml of the solution add 40 ml of *hydrochloric acid R* and titrate with 0.05M *potassium iodate* until the colour changes from red to yellow. Add 5 ml of

*chloroform R* and continue the titration, shaking vigorously, until the chloroform layer is decolourised.

1 ml of 0.05M *potassium iodate* is equivalent to 16.60 mg of KI.

**STORAGE**

Store protected from light.

Ph Eur

**Action and use**

Antithyroid.

**Potassium Nitrate**

(Ph Eur monograph 1465)

KNO<sub>3</sub>

101.1

7757-79-1

Ph Eur

**DEFINITION**

Potassium nitrate contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of KNO<sub>3</sub>, calculated with reference to the dried substance.

**CHARACTERS**

A white, crystalline powder or colourless crystals, freely soluble in water, very soluble in boiling water, practically insoluble in alcohol.

**IDENTIFICATION**

A. It gives the reaction of nitrates (2.3.1).

B. Solution S (see Tests) gives the reactions of potassium (2.3.1).

**TESTS****Solution S**

Dissolve 10.0 g in *carbon dioxide-free water R* prepared from *distilled water R* and dilute to 100 ml with the same solvent.

**Appearance of solution**

Solution S is clear (2.2.1) and colourless (2.2.2, *Method II*).

**Acidity or alkalinity**

To 10 ml of solution S add 0.05 ml of *bromothymol blue solution R1*. Not more than 0.5 ml of 0.01M *hydrochloric acid* or 0.01M *sodium hydroxide* is required to change the colour of the indicator.

**Reducible substances**

To 10 ml of solution S, add 0.5 ml of *dilute sulphuric acid R* and 2 ml of *zinc iodide and starch solution R*. The solution does not become blue within 2 min.

**Chlorides (2.4.4)**

If intended for ophthalmic use, it complies with the test for chlorides. Dissolve 2.5 g in *water R* and dilute to 15 ml with the same solvent. The solution complies with the limit test for chlorides (20 ppm).

**Sulphates (2.4.13)**

Dilute 10 ml of solution S to 15 ml with *distilled water R*. The solution complies with the limit test for sulphates (150 ppm).

**Ammonium (2.4.1)**

1 ml of solution S complies with the limit test (A) for ammonium (100 ppm). If intended for ophthalmic use, not more than 50 ppm of ammonium.

**Calcium (2.4.3)**

Dilute 10 ml of solution S to 15 ml with *distilled water R*. The solution complies with the limit test for calcium