Quality standards

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

Titanium Ointment

General Notices

DEFINITION

Titanium Ointment contains a large proportion of Titanium Dioxide with smaller quantities of titanium peroxide and titanium salicylate in a suitable basis containing 5.0% w/w of Dimeticone 350.

The ointment complies with the requirements stated under Topical Semi-solid Preparations and with the following requirements.

Content of total titanium, as titanium dioxide TiO,

22.5 to 27.5% w/w.

Content of titanium salicylate

2.70 to 3.30% w/w.

CHARACTERISTICS

A yellow, greasy ointment.

IDENTIFICATION

- A. Disperse 5 g in 20 mL of warm <u>dichloromethane</u> on a water bath and filter the resulting suspension (Whatman No 1 paper is suitable). Wash the residue with 50 mL of warm <u>dichloromethane</u> and transfer the residue to a 50 mL beaker with the aid of a few mL of <u>water</u>. Add carefully 10 mL of <u>sulfuric acid</u> and 5 g of <u>ammonium sulfate</u> and heat until a clear solution is obtained. Allow to cool, add sufficient <u>water</u> to produce about 30 mL and add 1 mL of <u>hydrogen</u> peroxide solution (20 vol). A bright orange colour is produced immediately.
- B. Disperse 5 g in 20 mL of warm <u>dichloromethane</u> on a water bath and filter the resulting suspension (Whatman No 1 paper is suitable). Wash the residue with 50 mL of warm <u>dichloromethane</u> and transfer the residue to a 50 mL beaker with the aid of a few mL of <u>water</u>. Mix well, filter again and add 0.05 mL of a 5% w/v solution of <u>iron(III) chloride hexahydrate</u> to the filtrate. A purple colour is produced immediately.

ASSAY

For TiO₂

Weigh 0.5 g into a tared silica crucible, heat gently over a small flame until the basis is completely volatilised or charred, increasing the rate of heating until almost all of the carbon has been removed. Heat at about 800° for about 30 minutes, allow to cool in a desiccator and weigh. Repeat the heating at 800° to constant weight. Calculate the content of total titanium as a percentage weight in weight using the formula:

$$\left(\frac{W_{\rm r}}{W_{\rm s}} \times 100\right) - 1.55$$

where

 W_{r} = weight of the residue,

W_s = weight of the sample

1.55 = percentage correction for contribution from Dimeticone 350.

For titanium salicylate

To 1.5 g in an iodine flask add 50 mL of 2M <u>ammonia</u> and heat gently until the solution boils. Add 25 mL of <u>0.0167M</u> <u>potassium bromate VS</u> and 10 mL of a 10% w/v solution of <u>potassium bromide</u>, add 20 mL of 7M <u>hydrochloric acid</u>, close the flask immediately and shake while cooling under running water. Allow the flask to stand for 30 minutes, protected from light. Add 10 mL of a 10% w/v solution of <u>potassium iodide</u>, shake and titrate the liberated iodine with 0.1M <u>sodium</u> <u>thiosulfate VS</u> using <u>starch solution</u>, added towards the end of the titration, as indicator. Each mL of <u>0.0167M potassium</u> <u>bromate VS</u> is equivalent to 2.818 mg of titanium salicylate.