UNGUENTUM WHITFIELD

CZ 151:2026

Whitfield’s ointment

DEFINITION

A hydrophobic suspension ointment with salicylic acid ($C_7H_6O_3$; M_r 138.12) and benzoic acid ($C_7H_6O_2$; M_r 122.12).

Content:

- *salicylic acid:* 2.7 % to 3.3 %;
- *benzoic acid:* 5.7 % to 6.3 %.

COMPOSITION AND PROCEDURE

Acidum salicylicum (*0366*) (90)	3.0 g
Acidum benzoicum (*0066*) (90)	6.0 g
Paraffinum liquidum (*0239*)	10.0 g
Adeps lanae (*0134*)	10.0 g
Vaselinum flavum (*1554*)	71.0 g

Levigate the mixture of salicylic acid and benzoic acid with liquid paraffin; while continuing to levigate, add wool fat and yellow petrolatum in portions.

CHARACTERISTICS

Appearance. Yellow soft homogeneous ointment with a characteristic odour.

IDENTIFICATION TESTS

A. Mix 1 g with 5 ml *chloroform R* in a beaker and mix the same amount in another beaker with 5 ml *water R.* The ointment does not dissolve in water but dissolves in chloroform (*hydrophobic ointment*). The chloroform solution is also used in test B.

B. Add 1 ml *acetic anhydride R* and 0.1 ml *sulphuric acid R* to the chloroform solution from identification test A and mix carefully; a dark green colour appears (*cholesterol*).

C. In a separating funnel, dissolve 5 g in 20 ml *medical benzine RN* and shake with a mixture of 20 ml *water R* and 10 ml *ethanol 96 % R.* Filter the aqueous layer, which is also used in identification test D. Add 0.05 ml *methyl red RS* to 5 ml of the filtrate and neutralise with *dilute sodium hydroxide RS* until the first yellow colour appears. Add 5 ml *water R* and 0.1 ml *ferric chloride RS1*; the solution turns reddish-purple (*salicylic acid*) and simultaneously a brown-red flocculent precipitate (*benzoic acid*) forms in the solution.

D. Mix 5.0 ml of the filtrate from identification test C with 5 ml *bromine water R* in a flask with a ground-glass stopper, close and leave to stand for 15 minutes. Filter and remove the excess bromine by gently boiling the filtrate until it becomes colourless. After cooling, add 0.05 ml *methyl red RS* and add *sodium hydroxide RS* dropwise until the first yellow colour appears. After adding 0.2 ml *ferric chloride RS1*, a light brown-red precipitate forms (*benzoic acid*).

DETERMINATION OF CONTENT

Benzoic acid. Dissolve 2.500 g in 20 ml *ether R* in a beaker, add 20 ml *ethanol 96% R* previously neutralised with *sodium hydroxide 0.1 mol/l VS* to 0.2 ml *thymol blue RS* until a yellow-green colour is obtained and titrate with the same volumetric solution to the same colour. The titrated solution is then used for the determination of salicylic acid.

1 ml *sodium hydroxide 0.1 mol/l VS* is equivalent to 12.21 mg $C_7H_6O_2$.

The percentage content of benzoic acid is calculated using the formula:

$$\left(\frac{a}{m} - \frac{y}{1.381}\right) \cdot 1.221$$

where:

a – volume of *0.1 mol/ VS sodium hydroxide* used, in millilitres;

m – mass of the preparation under test, in grams; *y* – percentage content of salicylic acid.

Salicylic acid. Quantitatively transfer the titrated liquid from the previous test to a separating funnel. Add 30 ml *sodium chloride R* solution (100 g/l) and mix. After clarification, separate the aqueous layer and wash the remainder in the separating funnel twice with 30 ml *sodium chloride R* solution (100 g/l). Combine the clarified aqueous layers and dilute with *water R* to 250.0 ml. Filter the solution and add 0.2 ml ammonium ferric sulphate solution (prepared by dissolving 10 g *ammonium ferric sulphate R* in 70 ml *water R* and 20 ml *dilute nitric acid RS*) to 10.0 ml clear filtrate in a 100 ml volumetric flask. Dilute the prepared mixture with *water R* to 100.0 ml and measure the absorbance (*2.2.25*) at the maximum at 525 nm against a compensation solution prepared by diluting 0.2 ml ammonium ferric sulphate solution with *water R* to 100.0 ml.

Simultaneously dissolve 30.0 mg *salicylic acid CRL* in 10 ml *ethanol 96 % R* in a 100 ml volumetric flask and dilute with *water R* to 100.0 ml. Add 0.20 ml ammonium ferric sulphate solution to 10.0 ml of this solution in a 100 ml volumetric flask, dilute with *water R* to 100.0 ml and measure the absorbance as described in the previous paragraph.

The percentage content of salicylic acid is calculated using the formula:

$$\frac{A\, m_s \cdot 250}{A_s\, m}$$

where:

A – absorbance of the sample solution of the preparation under test;

A_s – absorbance of the reference solution;

m —mass of the preparation under test used for the determination of benzoic acid, in grams; m_S– mass of *salicylic acid CRL*, in grams.

STORAGE

See article *Praeparata semisolida ad usum cutaneum* (*0132*).

SHELF LIFE

Six months when stored at 15 °C to 25 °C in plastic containers protected from light.