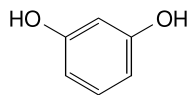


RESORCINOL

Resorcinolum



$C_6H_6O_2$
[108-46-3]

M_r 110.1

DEFINITION

Resorcinol contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of benzene-1,3-diol, calculated with reference to the dried substance.

CHARACTERS

A colourless or slightly pinkish-grey, crystalline powder or crystals, turning red on exposure to light and air, very soluble in water and in alcohol.

IDENTIFICATION

- A. Melting point (2.2.14): 109 °C to 112 °C.
- B. Dissolve 0.1 g in 1 ml of *water R*, add 1 ml of *strong sodium hydroxide solution R* and 0.1 ml of *chloroform R*, heat and allow to cool. An intense, deep-red colour develops which becomes pale yellow on the addition of a slight excess of *hydrochloric acid R*.
- C. Thoroughly mix about 10 mg with about 10 mg of *potassium hydrogen phthalate R*, both finely powdered. Heat over a naked flame until an orange-yellow colour is obtained. Cool and add 1 ml of *dilute sodium hydroxide solution R* and 10 ml of *water R* and shake to dissolve. The solution shows an intense green fluorescence.

TESTS

Solution S. Dissolve 2.5 g in *carbon dioxide-free water R* and dilute to 25 ml with the same solvent.

Appearance of solution. Solution S is clear (2.2.1) and not more intensely coloured than reference solution B₅ or R₅ (2.2.2, *Method II*) and remains so when heated in a water-bath for 5 min.

Acidity or alkalinity. To 10 ml of solution S add 0.05 ml of *bromophenol blue solution R2*. Not more than 0.05 ml of 0.1 M *hydrochloric acid* or 0.1 M *sodium hydroxide* is required to change the colour of the indicator.

Related substances. Examine by thin-layer chromatography (2.2.27), using *silica gel G R* as the coating substance.

Test solution. Dissolve 0.5 g of the substance to be examined in *methanol R* and dilute to 10 ml with the same solvent.

Reference solution. Dilute 0.1 ml of the test solution to 20 ml with *methanol R*.

Apply separately to the plate 2 µl of each solution. Develop over a path of 15 cm using a mixture of 40 volumes of *ethyl acetate R* and 60 volumes of *hexane R*. Allow the plate to dry in air for 15 min and expose it to iodine vapour. Any spot in the chromatogram obtained with the test solution, apart from the principal spot, is not more intense than the spot in the chromatogram obtained with the reference solution (0.5 per cent).

01/2008:0290 Pyrocatechol. To 2 ml of solution S add 1 ml of *ammonium molybdate solution R2* and mix. Any yellow colour in the solution is not more intense than that in a standard prepared at the same time in the same manner using 2 ml of a 0.1 g/l solution of *pyrocatechol R*.

Loss on drying (2.2.32). Not more than 1.0 per cent, determined on 1.00 g of powdered substance by drying in a desiccator for 4 h.

Sulphated ash (2.4.14). Not more than 0.1 per cent, determined on 1.0 g.

ASSAY

Dissolve 0.500 g in *water R* and dilute to 250.0 ml with the same solvent. To 25.0 ml of the solution in a ground-glass-stoppered flask add 1.0 g of *potassium bromide R*, 50.0 ml of 0.0167 M *potassium bromate*, 15 ml of *chloroform R* and 15.0 ml of *hydrochloric acid R1*. Stopper the flask, shake and allow to stand in the dark for 15 min, shaking occasionally. Add 10 ml of a 100 g/l solution of *potassium iodide R*, shake thoroughly, allow to stand for 5 min and titrate with 0.1 M *sodium thiosulphate*, using 1 ml of *starch solution R* as indicator.

1 ml of 0.0167 M *potassium bromate* is equivalent to 1.835 mg of $C_6H_6O_2$.

STORAGE

Store protected from light.

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corrected 6.0

RESTHARROW ROOT

Ononidis radix

DEFINITION

Whole or cut, dried root of *Ononis spinosa* L.

IDENTIFICATION

- A. The root is more or less flattened, twisted and branched, deeply wrinkled, brown and grooved longitudinally. The transversely cut surface shows a thin bark and a xylem cylinder with a conspicuously radiate structure. The fracture of the root is short and fibrous.
- B. Reduce to a powder (355) (2.9.12). The powder is light brown or brown. Examine under a microscope using *chloral hydrate solution R*. The powder shows the following diagnostic characters: brown fragments of cork composed of thin-walled polygonal cells; groups of thick-walled narrow fibres, often accompanied by a parenchymatous crystal sheath containing prisms of calcium oxalate; fragments of vessels with numerous small bordered pits; parenchymatous cells with single prisms of calcium oxalate. Examine under a microscope using a mixture of equal volumes of *glycerol R* and *water R*. The powder shows numerous simple, round starch granules, 5-10 µm in diameter.
- C. Thin-layer chromatography (2.2.27).

Test solution. To 1.0 g of the powdered drug (180) (2.9.12) add 15.0 ml of *methanol R* and boil under a reflux condenser for 30 min. Cool and filter.

Reference solution. Dissolve 10 mg of *resorcinol R* and 50 mg of *vanillin R* in 10 ml of *methanol R*.

Plate: TLC *silica gel F₂₅₄* plate *R*.

Mobile phase: *ethanol (96 per cent) R*, *methylene chloride R*, *toluene R* (10:45:45 V/V/V).