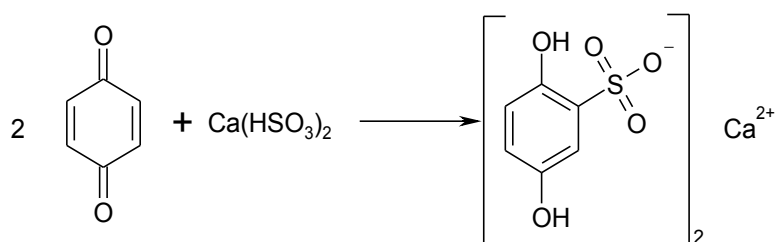


1. Calcium dobesilate

Systematic name: Calcium 2,5-dihydroxybenzenesulfonate

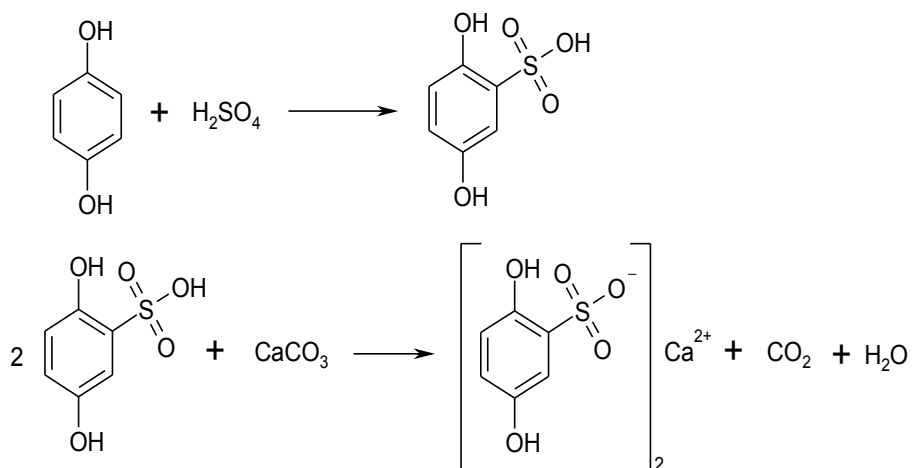
In industry, it is prepared by redox-substitution reaction of 1,4-benzoquinone with calcium hydrogensulfite:



Calcium hydrogensulfite is not commercially available due to its instability and is prepared by reaction of some suitable calcium(II) compound with sulfurous oxide in aqueous medium immediately before usage.

For purposes of our practical courses, an „alternative“ preparation procedure is more suitable. It consists of sulfonation of hydroquinone with sulfuric acid followed by neutralisation of thus formed 2,5-dihydroxybenzenesulfonic acid with calcium carbonate:

Scheme of preparation:



Chemicals:

hydroquinone [110.11] 1.0 g 0.009 mol

sulfuric acid 96% [98.08] 4 ml 0.075 mol

calcium carbonate [100.09] 12,5 g 0.124 mol

Procedure: Hydroquinone is added under stirring on a magnetic stirrer into a 50 ml flask with 96%

sulfuric acid at 45°C in a bath. Stirring is continued until all the amount of hydroquinone is dissolved (about 3 hours). After cooling to room temperature, the reaction mixture is poured under stirring into 120 ml of water in a beaker. Then calcium carbonate is gradually added under continuous stirring (slowly and carefully, a foam is formed!). The mixture is then shortly refluxed and still warm filtered by means of lowered pressure on a Büchner funnel (rounded paper filter of suitable diameter needed!). The filtrate is evaporated on a rotary vacuum evaporator until dryness. The residuum is then suspended in 25 ml of 96% ethanol and filtered through a normal paper filter (without support of vacuum). The filtrate is again evaporated until dryness. The residuum is the final product. The adequate portion of the residuum (approx. 0.2 g) is directly used for identity and purity confirmation by measurement of UV spectrum on a spectrophotometer HP 8453 according to the article Calcium dobesilate monohydrate (*Calcii dobesilas monohydricus*) of the actual European Pharmacopoeia. The specific absorption $A^{1\%}_{1\text{cm}}$ is calculated according to the formula

$$A^{1\%}_{1\text{cm}} = 40A / m,$$

where $A^{1\%}_{1\text{cm}}$ is specific absorption, A is absorbance measured in a maximum near 301 nm and m is the exact weight of calcium dobesilate in grams. The value of $A^{1\%}_{1\text{cm}}$ should range between 174-181. **If the product is stored until the next practical lesson, it must be kept in a suitable well closed vessel, protected against air moisture.** It is also suitable to dry it under reduced pressure at 40°C to the constant weight.