

COMPETITIVE PROBLEMS IN THE DRUG INDUSTRY 10391

6505-926-9055 (P. D. No. 3)

4.4 Tests.

4.4.1 Finished elixir.

4.4.1.1 Identity test (visible). Transfer 10 ml of elixir, accurately measured, to a 50-ml volumetric flask. Add purified water to volume and mix. Determine the absorbance of the clear liquid in a 1 cm quartz cell at about 530 mμ, with a suitable spectrophotometer and purified water as a blank, scanning from 750 mμ to 325 mμ. The absorbance shall be between 0.650 and 0.790.

4.4.1.2 Free p-aminophenol.

Equipment and Reagents.

Spectronic 20 or equivalent, equipped with 1 cm cells.

Sodium bicarbonate solution (NaHCO_3), 5% W/V aqueous.

Extracting solvent - Diethyl ether containing 1.5 ml of isoamyl alcohol per 100 ml.

Hydrochloric acid, 0.01N HCl.

Phenol solution, 1% aqueous.

Sodium carbonate solution (Na_2CO_3), 1N, approximately.

Sodium hypobromite solution - Add two drops of bromine water to 5 ml of 1N Na_2CO_3 and mix well. The solution should be slightly yellow. Prepare fresh.

Standard p-aminophenol solution. Dissolve 267.0 mg of p-aminophenol HCl (equivalent to 200 mg of p-aminophenol) in 1000 ml of purified water. Dilute 5.0 ml of standard solution to 100 ml with 0.01N HCl.

Procedure

Pipet 25.0 ml of sample into a 125 ml separatory funnel, allowing the pipet to drain for 10 to 15 minutes. Add 5 ml of 5% NaHCO_3 solution and mix. Add 50 ml of extracting solvent and shake vigorously for one minute, venting the funnel occasionally. Allow the layers to separate. Drain the lower aqueous layer into a small beaker and decant the ether layer into a 250 ml separatory funnel. Pour the aqueous layer back into the first separatory funnel and repeat the extraction process with two more 50 ml portions of extracting solvent. Combine all ether extracts in the second separatory funnel.