

10310 COMPETITIVE PROBLEMS IN THE DRUG INDUSTRY

6505-890-2012 (P. D. No. 3)

4.1 Tests.

4.1.1 Assays for phenylephrine hydrochloride, chlorpheniramine maleate, and codeine phosphate in the finished syrup shall be conducted in accordance with the method specified in the article titled "Analysis of Combinations Containing Phenylephrine in Liquid Dosage Forms," by K. O. Montgomery, P. V. Jennings, and M. Y. Weinswig, which appears in the Journal of Pharmaceutical Sciences, Vol. 56, No. 3, page 393, March 1967.

As an alternate, the method may be modified to provide for the elution of phenylephrine with 0.5N hydrochloric acid in purified water, the codeine with 1N hydrochloric acid in (1:1) methanol:purified water, and the chlorpheniramine with 5N hydrochloric acid in (1:1) methanol:purified water. Absorbances shall be determined using the base line technique.

In addition, the method may be further modified by the use of an anion column between the reservoir and the cation column.

4.1.2 Assay for glyceryl guaiacolate in the finished syrup.

Standard.

Accurately weigh approximately 40 mg of Glyceryl Guaiacolate N.F. Reference Standard into a 250-ml volumetric flask and dilute to volume with chloroform. Pipet 20 ml of this solution into a 100-ml volumetric flask and dilute to volume with chloroform.

Procedure.

Pipet 2 ml, using a "to contain" pipet, of syrup (sample) into a 125-ml separator. Rinse the pipet with purified water. Add purified water to the separator until the volume is approximately 45 ml. Add 10 grams of anhydrous sodium sulfate and shake vigorously to dissolve the salt. Add 5 ml of 10 percent sodium hydroxide solution and mix well. Wash the aqueous layer thoroughly with 50 ml isooctane (A.R.). Transfer the aqueous layer to a second 125-ml separator. Wash the isooctane with 2 ml of purified water. Add this wash to the second separator. Discard the isooctane. Extract the aqueous layer with five 40-ml portions of chloroform (A.R.). Filter the chloroform through Whatman No. 1 paper into a 250-ml volumetric flask. Rinse the paper and funnel with chloroform, dilute to volume, and mix. Pipet 20 ml of this solution into a 100-ml volumetric flask, q.s. with chloroform, and mix well.