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论文

原儿茶醛分析方法的研究——Ⅱ.四季青和丹参中原儿茶醛的测定

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摘要:

应用间苯三酚和原儿茶醛在浓硫酸存在下的显色反应,结合薄层层析法建立了测定四季青和丹参中原儿茶醛的分析方法。本法简便准确,原儿茶醛在生药中的含量为万分之几而其测定结果的变更系数小于2%。

关键词:

STUDIES ON THE ANALYSIS OF PROTOCATECHUALDEHYDE——Ⅱ. DETERMINATION OF PROTOCATECHUALDEHYDE IN *ILEX CHINENSIS* SIMS AND *SALVIA MILTIORRIZA* BGE

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Abstract:

A method for the determination of protocatechu aldehyde in *Ilex chinensis* Sims and *Salvia miltiorrhiza* Bge was developed by using phloroglucinol—sulfuric acid as the colorimetric reagent and TLC as the separation means. The method is steady and reproducible: Seven determinations of protocatechualdehyde in crude drug gave a result of $0.0547\pm8.54\times10^{-4}$% with a variation coefficient of 1.56%.The analytical procedure is as follows: Put 0.2～0.3 g pulverized sample of crude drug in a 20 ml glass-stoppered test-tube. After having soaked the sample with 3 ml H_2O, place the test tube in a boiling waterbath for an hour and a half. Cool, add 1ml of saturated sodium chloride solution and 10.0ml of ethyl ether, stopper tightly (wet the stopper first)and shake the mixture for about three minutes. After the supernatant solution has cleared up, transfer 5.0 ml into a glass-stoppered pear-shaped flask and add several small chips of pumice. Put the flask in a warm waterbath to evaporate the solvent. Cool, add 0.20 ml of 1:1 MeOH-$CHCl_3$, stopper tightly, shake the flask until the residue has been completely dissolved. Put 20～30 μl of this solution on a silica gel G plate (5.5×20 cm) in the form of a line, at the same time spot a pure protocatechualdehyde sample as the reference standard and develop with $CHCl_3$-MeOHHCOOH (90:8:2). Then, take out the plate from the chromatographic chamber and let it stand for about half an hour to allow the dark band of protocatechualdehyde to show up. Scrape this band into a 10 ml glass-stoppered centrifuge tube, add 5.0ml of 1:1 cold mixed solution of 2% phloroglucinol solution (in ethanol)and concentrated sulfuric acid. Shake the mixture vigorously for a moment and put it together with a reagent blank in a waterbath at 50℃ for 5 minutes. Cool with cold water and centrifuge the mixture for several minutes. Determine the absorbance of the supernatant liquid at 496 nm in an 1 cm cell against the reagent blank. Calculate the content of protocatechualdehyde from a calibration curve.

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