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论文

薄层层离法在研究天然化合物中的应用 Ⅱ.麦角新碱、麦角胺、麦角毒碱的定量

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摘要:

本文研究了应用无粘合剂氧化鋁薄层层离和光电比色相結合的方法,测定麦角中三种主要生物碱的含量。回收率試驗表明,本方法具有較高的准确性,如麦角新碱的平均回收率为97.3%,标准誤差±1.7%;麦角胺的平均回收率为96.2%,标准誤差±2.2%;麦角毒碱的平均回收率为99.3%,标准誤差±0.9%。加量試驗的結果表明,本方法可以应用于麦角中三种主要生物碱的含量测定。作者用此方法测定了披碱草、老芒麦、高滨麦三种野生麦角和二种麦角菌发酵物中各种麦角生物碱的含量。操作簡便,有机溶剂用量很少。

关键词:

APPLICATION OF THIN LAYER CHROMATOGRAPHY IN THE STUDY OF NATURAL PRODUCTS——Ⅱ.QUANTITATIVE DETERMINATION OF ERGOMETRINE ERGOTAMINE AND ERGOTOXINE

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Abstract:

Application of the thin layer chromatographic method to the determination of ergometrine, ergotamine and ergotoxine in ergot has been studied. One gram of defatted ergot was accurately weighed and macerated with a mixture of 10 ml chloroform-methanol-ammonia (90:9:1) for 24 hours in a brown flask (50 ml volume). Five ml of the extract were pipetted into an Erlenmeyer flask (10 ml volume), placed in a vacuum desiccator and pumped to dryness at room temperature. The residue was dissolved in 0.25ml methanol, 0.1 ml being applied onto a 0.05×4.5×19 cm alumina layer and developed with a mixture of benzene-chloroform-absolute alcohol (7:3:0.5). Fluorescent spots of ergometrine, ergotamine and ergotoxine were located under UV light and separately transferred with a collector into reagent tubes. To each of these were added 0.5 ml ethanol, 1.5 ml 0.2*N* sulphuric acid, and 4ml van Urk's reagent. After shaking and standing for half an hour, the mixture was centrifuged and the clear blue solution determined colorimetrically. Analysis of an ergot sample, repeated six times, gave an average of 0.0255% for ergometrine, 0.0178% for ergotamine and 0.0143% for ergotoxine. Recovery of pure ergot alkaloids and experiments with added alkaloids showed high accuracy by this method. The procedure is simple and requires little organic solvents.

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