

研究论文

制备型液相色谱法分离纯化3-甲基吡啶光氯化产物中的2-氯-5-三氯甲基吡啶

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摘要 利用制备型高效液相色谱从3-甲基吡啶光氯化产物中分离纯化得到2-氯-5-三氯甲基吡啶, 对制备色谱的洗脱方式、洗脱剂组成及浓度、进样量等参数进行了优化。使用的制备柱为Zorbax-C18柱, 以乙腈-水为流动相, 采用速度梯度洗脱方式进行洗脱, 用二极管阵列检测器在240 nm波长下检测, 进样体积为100 μL。该方法的制备回收率为82.7%, 相对标准偏差为4.0% (n=5), 产品纯度为99.01%。

关键词 [制备型液相色谱](#) [速度梯度洗脱](#); [2-氯-5-三氯甲基吡啶](#) [3-甲基吡啶](#); [光氯化产物](#)

分类号

Separation and Purification of 2-Chloro-5-Trichloromethylpyridine from Products of Photochemical Chlorination of 3-Methylpyridine Using Preparative Liquid Chromatography

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Abstract

A preparative high performance liquid chromatographic technique was used to separate and purify 2-chloro-5-trichloromethylpyridine from the products of photochemical chlorination with 3-methylpyridine as raw material. The elution mode, the flow rate of the mobile phase and injection volume were optimized. The optimum operation parameters were selected as follows: Zorbax-C18 reversed-phase column with the flow rate gradient elution of 30% acetonitrile in water as the mobile phase (gradient elution program: 0-8 min at 4 mL/min, 8-14 min at 10 mL/min), and a diode array detector with wavelength set at 240 nm, injecting volume of 100 μL. The average recovery rate was 82.7% and relative standard deviation (RSD) was 4.0% (n=5). High purity (99.01%) product was obtained. The effective and accurate method has been successfully applied to the preparation of pure 2-chloro-5-trichloromethylpyridine.

Key words

[preparative liquid chromatography](#) [flow-rate gradient elution](#) [2-chloro-5-trichloromethylpyridine](#) [3-methylpyridine](#) [photochemical chlorination product](#)

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