

技术与应用

## 顶空液相微萃取-气相色谱法测定盐酸雷尼替丁中二氯甲烷和三氯甲烷的残留量

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**摘要** 采用顶空液相微萃取与气相色谱联用技术测定雷尼替丁中二氯甲烷和三氯甲烷的残留量。自制了萃取液保护装置。考察了萃取溶剂的种类、萃取时间、萃取温度、萃取液的体积对二氯甲烷和三氯甲烷萃取效果的影响。以正十三烷为萃取剂,在60 °C下萃取30 min,萃取液滴体积2 μL。二氯甲烷含量在1~10 μg/g范围内与色谱峰高呈线性关系,相关系数( $r^2$ )为0.9733;三氯甲烷含量在1~10 μg/g范围内与色谱峰高呈线性关系, $r^2$ 为0.9724。二氯甲烷和三氯甲烷的最低检出限分别为0.0273 μg/g和0.0410 μg/g,加标回收率分别为93.6%~102%和98.1%~103%。方法简便易行,测定结果准确。

**关键词** [液相微萃取](#) [气相色谱法](#) [二氯甲烷](#) [三氯甲烷](#) [盐酸雷尼替丁](#)

## Determination of dichloromethane and trichloromethane residues in ranitidine hydrochloride by headspace liquid phase microextraction coupled with gas chromatography

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### Abstract

A method for the determination of residual dichloromethane and trichloromethane in ranitidine hydrochloride by headspace liquid phase microextraction coupled with gas chromatography (GC) was developed. A homemade device was used to protect the organic drop. The effects of the nature of extraction solvent, extraction time, extraction temperature and microdrop volume on the extraction efficiency were investigated separately. The optimal experimental conditions were as follows: 2 μL of n-tridecane as extraction solvent, 30 min of extraction time, 60 °C of extraction temperature. The correlation coefficients of linear calibration curve were 0.9733 and 0.9724 within the concentration ranges of dichloromethane (1~10 μg/g) and trichloromethane (1~10 μg/g), respectively. The detection limits of dichloromethane and trichloromethane were 0.0273 μg/g and 0.0410 μg/g, respectively, the relative standard deviations were lower than 4.36% and 5.89%, and the recoveries of the method were 93.6%~102% and 98.1%~103%, respectively. The method is simple and reliable.

**Key words** [liquid phase microextraction](#) [gas chromatography \(GC\)](#) [dichloromethane](#) [trichloromethane](#) [ranitidine hydrochloride](#)

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