

固相微萃取-气相色谱法测定工业苯酚中的2-甲基苯并呋喃和2,4-二苯基-4-甲基-1-戊烯

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Determination of 2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene in industrial phenol by solid-phase microextraction coupled with gas chromatography

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摘要 建立了工业苯酚中有机杂质的固相微萃取-气相色谱(SPME-GC)分析方法。实验考察了SPME萃取温度和萃取时间的影响,同时也优化了热解吸时间。优化后的萃取温度为20℃,萃取时间为10 min,热解吸时间为30 s。使用此法对工业苯酚样品中的两种主要有机杂质进行了分析检测,结果表明:2-甲基苯并呋喃和2,4-二苯基-4-甲基-1-戊烯分别在0.05~1.06 mg/L和0.05~0.99 mg/L范围内线性关系良好(r^2 分别为0.990和0.992),检出限分别为0.5和1.6 $\mu\text{g/L}$ 。在0.1 mg/L的添加水平下,2-甲基苯并呋喃和2,4-二苯基-4-甲基-1-戊烯的回收率分别为104%和113%。该方法具有简单、快速、灵敏度高等优点,适合于工业苯酚中这两种主要痕量有机杂质的准确定量分析。

关键词: 固相微萃取 气相色谱 工业苯酚 2-甲基苯并呋喃 2,4-二苯基-4-甲基-1-戊烯

Abstract: An analytical method based on solid-phase microextraction (SPME) coupled with gas chromatography for the determination of organic impurities (2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene) in industrial phenol was developed. The SPME parameters such as extraction temperature, extraction time, and the desorption time were optimized. The optimized parameters were as follows: extraction temperature was 20 $^{\circ}\text{C}$, extraction time was 10 min, and desorption time was 30 s. The results demonstrated that the linearities of calibration curves were good in the ranges of 0.05~1.06 mg/L and 0.05~0.99 mg/L with the correlation coefficients of 0.990 and 0.992 for 2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene, respectively. The limits of detection (LODs) were 0.5 and 1.6 $\mu\text{g/L}$ for 2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene, respectively. The relative recoveries were 104% and 113% for 2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene at the spiked level of 0.1 mg/L with triplicate determination. The method is simple, rapid and sensitive for the quantitative analysis of trace organic impurities in industrial phenol.

Keywords: solid-phase microextraction gas chromatography industrial phenol 2-methyl benzofuran 2,4-diphenyl-4-methyl-1-pentene

Received 2010-07-08; published 2010-10-28

Fund:

吉林省科技发展计划项目(No. 20095022)和中国石油天然气股份有限公司吉林石化分公司科技计划项目(No. 2008-12117).

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引用本文:

娄大伟1, 孙秀云1*, 杨积学2, 李自恩1, 牛春芳1, 赵飞1, 姜国玉2, 胡春福2, 牛志蒙2, 金辉2.固相微萃取-气相色谱法测定工业苯酚中的2-甲基苯并呋喃和2,4-二苯基-4-甲基-1-戊烯[J] 色谱, 2010,28(10): 989-992

LOU Dawei, SUN Xiuyun, YANG Jixue, LI Zien, NIU Chunfang, ZHAO Fei, JIANG Guoyu, HU Chunfu, NIU Zhimeng, JIN Hui.Determination of 2-methyl benzofuran and 2,4-diphenyl-4-methyl-1-pentene in industrial phenol by solid-phase microextraction coupled with gas chromatography[J] Chinese Journal of Chromatography, 2010,28(10): 989-992

链接本文:

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