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研究论文

分散液-液微萃取-气相色谱/质谱联用法测定机械加工水基切削液及其废水中的三氯苯

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摘要 建立了以丙酮为分散剂、氯苯为萃取剂，采用分散液-液微萃取、气相色谱/选择离子质谱联用测定机械加工水基切削液及其废水中三氯苯的方法。该方法与顶空萃取、液-液萃取和固相萃取结合气相色谱/电子捕获检测法相比，具有线性范围广、富集倍数高、重现性好、操作简便、干扰小等优点。样品中三氯苯的加标回收率为94.7%~104.3%，相对标准偏差为2.3%～7.8%。三氯苯的3种同分异构体1,3,5-，1,2,4-和1,2,3-三氯苯的检出限分别为2.0，6.0和3.0 μg/L。重点探讨了萃取剂和分散剂的种类、体积、萃取时间和盐效应等对三氯苯萃取效率的影响，优化了萃取条件。考察了机械加工水基切削液中常用的添加剂对检测结果的影响，结果表明1.0%的亚硝酸钠和聚乙二醇对三氯苯的检测基本无影响。采用该方法对4种实际样品中的三氯苯进行了测定，其中两个样品中含有三氯苯，质量浓度范围为0.15～1.67 mg/L。

关键词 分散液-液微萃取；气相色谱/质谱联用；三氯苯；水基切削液；机械加工废水

Determination of trichlorobenzenes in water-based cutting fluids and wastewater of machining using dispersive liquid-liquid microextraction-gas chromatography/mass spectrometry

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Abstract

The determination of trichlorobenzenes (TCBs) in water-based cutting fluids and wastewater of machining has been carried out. A gas chromatography/mass spectrometry (GC/MS) method with selected ion monitoring (SIM) mode was employed. The target analyte was extracted from the matrix using dispersive liquid-liquid microextraction. Comparing with gas chromatography/electronic capture detection (GC/ECD) coupled with traditional sample preparation procedures, e.g. head-space extraction, liquid-liquid extraction and solid-phase extraction, the present method was accurate with broader linear range, better enrichment property, better replicability, easier to be operated and less interference. Overall recoveries were 94.7%-104.3% with the relative standard deviations （RSDs） of at 2.3%-7.8%. The detective limits for 1,3,5-, 1,2,4- and 1,2,3-trichlorobenzene were 2.0, 6.0 and 3.0 μg/L, respectively. The parameters, such as the nature and volume of extraction solvent, dispersive solvent, extraction time and salt effect, were studied and optimized. Some important factors, e.g., the concentration of common used additives in water-based cutting fluids, which may affect the recoveries and replicabilities for the determination of trichlorobenzenes, have been investigated. The result showed that no significant effects have been observed when the concentrations of NaNO2 and polyethylene glycol （PEG） were up to 1.0 %. The present method has been applied for the determination of the trichlorobenzenes in 4 real samples. The result showed that two of them were found to contain these trichlorobenzenes. The TCBs in the samples were 0.15-1.67 mg/L.

Key words dispersive liquid-liquid microextraction gas chromatography/mass spectrometry (GC/MS) trichlorobenzenes (TCBs) water-based cutting fluids wastewater of machining

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