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离子液体-顶空气相色谱法检测酮康唑中的多种高沸点有机残留溶剂

ILs as Headspace Matrix Medium for Determining High-boiling Organic Residual Solvent in Ketoconazole
HS-GC

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中文摘要:

目的 建立酮康唑原料药中有机溶剂残留量检测的高灵敏方法, 初步探索有机溶剂与离子液体(ILs)之间相互作用规律, 为选: 适的ILs作为顶空溶剂进行残留测定提供依据。方法 精密称取原料药0.01 g, 置10 mL顶空瓶中, 精密加入1 mL ILs, 密封, 微波功 0 W辐射75 s溶解后, 置110 °C恒温箱中平衡30 min, 抽取顶部空气1 mL, 进气相色谱仪(GC)分析检测, 考察并优化顶空溶剂。采用 毛细管柱(100%二甲基聚硅氧烷, 0.53 mm×60 m, 5 μm), FID检测器, 程序升温。结果 乙醇、二氯甲烷、乙酸乙酯、正丁醇、吡 N,N-二甲基甲酰胺、二甲基亚砷分离度良好, 分别在1.25~200, 1.50~24.0, 12.5~200, 12.5~200, 0.500~8.00, 2.20~3.52, 12.5 mg·L⁻¹内线性关系良好, 平均回收率在89.8%~98.2%内, RSD均<4.0%。对于乙醇, ILs的顶空效率随着阳离子烷基链的增长而增加, 乙酸乙酯、DMF和DMSO, ILs的顶空效率随阳离子烷基链的增长而降低, 而阴离子对ILs的顶空效率影响相对较小, 最终确定[Bmim][I 为最佳顶空溶剂。结论 该方法简单、快速、灵敏度高, 适用于同时测定酮康唑原料药中多种有机溶剂残留, 为药物中高沸点有机残 剂的检测提供了一种新手段。对于质子性溶剂, ILs的顶空效率随阳离子极性的减小而增加, 对于具有亲质子能力的非质子性溶剂, 的顶空效率随阳离子极性的减小而降低, 而阴离子的极性对ILs顶空效率影响较小, 为选择合适的ILs作为顶空溶剂进行残留测定提 考依据。

英文摘要:

OBJECTIVE To establish a sensitive method to determine the organic residual solvents in Ketoconazole. The interaction between organic solvents and ILs was preliminarily explored, intending to provide a basis for choosing suitable ILs as headspace solvent to determinate the residual solvents. METHODS 0.01 g of ketoconazole were put in 10 mL headspace vial, followed by spiking 1 mL of ILs. The headspace vial was sealed and radiated under 150 W of microwave power for 75 s to accelerate the dissolution of Ketoconazole. The sample solution was maintained at the equilibration temperature of 110 °C for 30 min and a volume of 1 mL headspace gas was directly injected into GC for analysis. A phenomenex ZB-1 capillary column (0.53 mm×60 m) with 5.00 μm film thickness was utilized for chromatographic separation of the solvents with FID as detector. A temperature programme was also employed. RESULTS excellent separation of ethanol, dichloromethane, ethyl acetate, butyl alcohol, pyridine, DMF and DMSO was achieved. The calibration curve was linear in the range of 1.25-200 mg·L⁻¹ for ethanol, 1.50-24.0 mg·L⁻¹ for dichloromethane, 12.5-200 mg·L⁻¹ for ethyl acetate, 12.5-200 mg·L⁻¹ for butyl alcohol, 0.500-8.00 mg·L⁻¹ for pyridine, 2.20-3.52 mg·L⁻¹ for DMF and 12.5-200 mg·L⁻¹ for DMSO. All the average recovery were limited in 89.8%-98.2% and RSD were less than 4.0%. For ethyl, the headspace efficiency of ILs increased with the growth of cation alkyl chain. For ethanol, DMF and DMSO, the headspace efficiency of ILs decreased with the growth of cation alkyl chain. However, the anion of ILs had minor impact on headspace efficiency. Ultimately, [Bmim][PF6] was chosen as the best headspace solvent. CONCLUSION

proposed method is easy, fast and sensitive. It is suitable for the simultaneous determination of ethanol, dichloromethane, ethyl acetate, butyl alcohol, pyridine, DMF and DMSO in Ketoconazole, providing a new mean for the detection of residual solvent with high-boiling point. The headspace efficiency of ILS increased with the decrease of the cationic polarity for protic solvents and reduced with the decrease of cation polarity for aprotic solvents with nucleophilic ability, while the polarity of the anion has little influence on headspace efficiency. The results are