

固相萃取-离子色谱法测定甘蔗糖蜜及糖蜜酒精废液中的非氮有机酸和无机阴离子

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Determination of nonnitrogenous organic acids and inorganic anions in sugarcane molasses and molasses alcohol waste by ion chromatography with solid-phase extraction

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摘要 建立了固相萃取-离子色谱测定甘蔗糖蜜及糖蜜酒精废液中乙酸、乳酸、琥珀酸、苹果酸、酒石酸、草酸、富马酸、柠檬酸、乌头酸等非氮有机酸和盐酸根、硫酸根、磷酸根等3种无机阴离子的方法。样品稀释液经强阴离子(SAX)固相萃取小柱净化除去糖类和色素等干扰基质,再用稀KOH溶液洗脱,经0.45 μm水膜过滤后,用IonPac AS15阴离子分离柱、KOH溶液梯度淋洗-抑制电导检测分离分析。考察了固相萃取小柱对待测离子的保留和洗脱条件。实验结果表明,除乙酸和乳酸的分离不完全、苹果酸与琥珀酸的组分重叠外,其余组分可达到完全分离,被测组分的浓度与其峰高在一定的范围呈良好的线性关系,检出限均低于0.20 mg/L,相对标准偏差(RSD)小于6.7%。测定了2种甘蔗糖蜜和1种糖蜜酒精废液中有有机酸及无机阴离子,结果满足检测的要求,样品中各组分的加标回收率为94%~109%。

关键词: 固相萃取 离子色谱 非氮有机酸 无机阴离子 甘蔗糖蜜 糖蜜酒精 废液

Abstract: A method was established for the determination of main nonnitrogenous organic acids (including acetic, lactic, succinic, maleic, tartaric, oxalic, fumaric, citric, and aconitic acids) and three inorganic anions (Cl⁻, SO₂⁻⁴ and PO₃⁻⁴) in sugarcane molasses and molasses alcohol waste by ion chromatography with solid-phase extraction. The diluted sample solution was purified by strong anion exchange (SAX) solid-phase extraction (SPE) in a small packed column to remove most of sugar, pigment and other interfering matrices. The eluate obtained from the column was rinsed with diluted KOH solution and filtered through a 0.45 μm inorganic membrane, finally separated on an anion column of IonPac AS15 with the gradient elution of KOH solution and determined with a suppressed conductivity detector. The pretreatment and procedures of SPE for separating organic acids and inorganic anions from their matrix were investigated. The detection limits were less than 0.20 mg/L. The relative standard derivations were less than 6.7%. The organic acids and inorganic anions in three real samples (two sugarcane molasses samples and an alcohol waste sample) were determined, the recovery ranges were from 94% to 109%. The method shows good precision and linearity.

Keywords: solid-phase extraction (SPE) ion chromatography (IC) nonnitrogenous organic acids inorganic anions sugarcane molasses molasses alcohol waste

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