色谱 » 2011, Vol. 29 » Issue (01) :75-78 DOI: 10.3724/SP.J.1123.2011.00075

高效阴离子交换色谱-脉冲安培检测法定量测定低聚木糖样品中的低聚木糖

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Quantitative determination of xylo-oligosaccharides in xylo-oligosaccharide products with high performance anion-exchange chromatography coupled with pulsed amperometric detection

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摘要 建立了低聚木糖样品中的木二糖至木六糖等低聚木糖的高效阴离子交换色谱定量测定方法,并根据低聚木糖的聚合度与色谱保留时间的线性关系,对木七糖和木八糖的保留时间进行预测。采用CarboPacTM PA200阴离子交换柱(3 mm×250 mm),以醋酸钠和氢氧化钠为淋洗液进行二元梯度洗脱,脉冲安培法进行检测。结果表明,木二糖至木六糖在0.804～8.607 mg/L质量浓度范围内的线性关系良好,检出限为0.064～0.111 mg/L,定量限为0.214～0.371 mg/L。将该方法用于低聚木糖产品的检测,3个添加水平的加标回收率为84.29%～118.19%,相对标准偏差(n=3)为0.44%～14.87%。结果表明该方法适用于低聚木糖产品中有效成分的快速、高效分离和定量测定。

关键词： 高效阴离子交换色谱 脉冲安培检测 低聚木糖

Abstract： A method for the analysis of xylo-oligosaccharides(XOS) in xylo-oligosaccharide products, including xylobiose, xylotriose, xylotetraose, xylopentaose and xylohexaose, was developed using high performance anion-exchange chromatography coupled with pulsed amperometric detection (HPAEC-PAD). The retention times of xyloheptaose and xylooctaose were calculated according to the linear relationship between the retention time and the polymerization degree. The separation was performed on a CarboPacTM PA200 column (250 mm×3 mm) with a gradient elution of NaOH-NaOAc as the mobile phase. The calibration curves showed good linearity for the xylo-oligosaccharides in the range of 0.804~8.607 mg/L. The detection limits (LODs) and the quantification limits (LOQs) were 0.064~0.111 mg/L and 0.214~0.371 mg/L, respectively. Under the optimized conditions, the recoveries of xylo-oligosaccharides at three different spiked levels ranged from 84.29%~118.19%, with the relative standard deviations (RSDs, n=3) of 0.44%~14.87%. This method is fast and accurate for the quantitative analysis of the xylo-oligosaccharide products.

Keywords： high performance anion-exchange chromatography (HPAEC) pulsed amperometric detection (PAD) xylo-oligosaccharides

Received 2010-09-14; published 2011-01-21

Fund:

国家自然科学基金项目(No. 31070514)、江苏省高校自然科学重大基础研究项目(No. 08KJA220002)和江苏省自然科学基金项目(No. BK2009690).

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引用本文:

范丽, 徐勇*, 连之娜, 勇强, 余世袁.高效阴离子交换色谱-脉冲安培检测法定量测定低聚木糖样品中的低聚木糖[J] 色谱, 2011,V29(01): 75-78

FAN Li, XU Yong*, LIAN Zhina, YONG Qiang, YU Shiyuan.Quantitative determination of xylo-oligosaccharides in xylo-oligosaccharide products with high performance anion-exchange chromatography coupled with pulsed amperometric detection[J] Chinese Journal of Chromatography, 2011,V29(01): 75-78

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