

研究论文

10° 锥角台锥型制备液相色谱柱的放大研究

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摘要 将10° 锥角台锥型液相色谱柱放大至150 mm长、入口直径54 mm、出口直径27 mm, 容积为200 mL, 填料为粒径40~75 μm、孔径11 nm的C18球形硅胶。流动相在锥型柱内呈现塞子状流形。系统地评价了该柱的分离性能, 结果表明: 在最佳流速为6 mL/min时, 以萘峰计, 锥型柱的折合理论塔板高度为2.11, 柱效下降10%时的样品质量和体积载样量分别为2.1 mg和1.7 mL, 与同长度同体积圆柱型柱相比, 柱效提高了20%, 质量载样量提高了16%以上, 体积载样量提高了19%以上。当进样质量由2.4 mg增加到12 mg时, 对羟基苯甲酸乙酯峰与对羟基苯甲酸丁酯峰的分度度(R_s2)由2.14降到1.71, 对羟基苯甲酸丁酯峰与萘峰的分度度(R_s3)由2.91降到2.52; 当进样体积由3 mL增加到19 mL, R_s2 由2.23降到1.28, R_s3 由2.95降到2.30, 但此时的色谱峰峰形仍然高度对称, 没有拖尾, 有利于从基质中制备分离微量组分。实验结果表明锥型液相色谱柱将具有广泛的应用前景。

关键词 [制备液相色谱](#) [台锥型色谱柱](#) [放大](#)

Scale-up of conical column with 10° opening angle as preparative liquid chromatographic column

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Abstract

A preparative scale liquid chromatographic column with the conical shape of 10° opening angle was constructed and evaluated. The column was designed with the inlet/outlet diameters of 54/27 mm, the column length of 150 mm and the column volume of 200 mL, and packed with the spherical C18 bonded silica with the particle size of 40~75 μm and the aperture of 11 nm. The mobile phase in the conical column showed a plug like flow profile and plug like chromatographic band shape. For naphthalene, the reduced plate height was about 2.11; the maximum sample load was 2.1 mg or 1.7 mL (10% reduction of plate number), which is 20%, 16% and 19% higher than that of cylindrical one of the same length and volume. As the injection mass increased from 2.4 mg up to 12 mg, the resolution of ethyl paraben/butyl (R_s2) reduced from 2.14 down to 1.71, and the butyl paraben/naphthalene (R_s3) from 2.91 down to 2.52; the injection volume increased from 3 mL up to 19 mL, R_s2 reduced from 2.23 down to 1.28, and R_s3 from 2.95 down to 2.30, while the peaks were still in symmetric shape without tailing. This characteristic of the column shall benefit for the separation of trace components from matrix. This demonstrated the conical shaped preparative columns would have a broad practical applicability for obtaining pure compounds.

Key words [preparative liquid chromatography](#) [conical chromatographic column](#) [scale-up](#)

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