

Abstract

Non-silica based materials in drug analysis IV.

Diploma thesis

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Sample preparation is an important step before the analysis itself. It enables removal of the ballast components, which could interfere with an analyte or even disable the analysis. Solid phase extraction (SPE) belongs among the most popular sample preparation techniques. Silica is the most often used sorbent for SPE, however new, more durable sorbents with different selectivity have appeared. This thesis focuses on employment of zirconium dioxide and its ability to retain analytes having Lewis base character via ligand exchange. We tested to use zirconium dioxide in extraction of indomethacin as a Lewis base from suppositories. The influence of conditioning solvents with different polarity and extractions solvents was investigated. 10% acetonitrile in methylene chloride was found to be the best solvent for conditioning of zirconia. Methanolic ammonia solution was used as eluent. After some experiments the concentration and the volume of the eluent was set at 0.25M NH₃ in MeOH and 3 mL. Diclofenac was used as the internal standard. The recovery for sorbent after its regeneration was found to be 83 % for the standard of indomethacin and 72 % for indomethacin in a suppository. The adsorption of the analyte on new, non-regenerated sorbent was stronger, therefore 20% acetonitrile in methylene chloride was found to be better for conditioning. The recovery was found to be 80 % for the standard of indomethacin and 84 % for suppository. The amount of the suppository mass was proven not to interfere with the extraction. Linearity of the extraction was proven.

Key words: SPE, ZrO₂, indomethacin