

Detection of migration of phthalates from agglomerated cork stoppers using HPLC-MS/MS. Raquel Sendón, Ana Sanches-Silva, Juana Bustos, Patricia Martín, Nuria Martínez and M^a Eugenia Cirugeda. *Journal of Separation Science*, Volume 35, Issue 10-11, June 2012, Pages: 1319–1326.

Abstract

Agglomerated stoppers are manufactured from natural cork granules and adhesives. Esters, such as phthalates and adipates, are commonly used in adhesives at concentrations of typically 2–5%. Because of this, and regarding consumer safety, it is necessary to ensure that these compounds do not migrate into the beverage where the cork stopper is used. A reversed-phase high performance liquid chromatography method with tandem mass spectrometry detection is developed for the separation of nine phthalates into 12% ethanol, used as simulant of alcoholic beverages. The chromatographic separation was carried out with a Luna C18 (2) HSTcolumn (50 × 3.0 mm, 2.5 μm) with a gradient elution of water/methanol with 0.1% acetic acid at 300 μL min⁻¹. The method was validated for four selected phthalates: di-butylphthalate, di-isononylphthalate, di-isodecylphthalate, and butyl-benzyl phthalate, with recoveries ranging between 95% and 112% and intralaboratory precision (RSD) between 5 and 14%, depending on the phthalate. The lowest quantification limit, 0.15 mg kg⁻¹, was achieved for di-butylphthalate. Nevertheless, in all cases, the limits obtained guarantee the method utility if restriction limits set in Commission Regulation No 10/2011 for plastic materials are taken into account.