

Method development and validation for phenol and nitrophenols in tap water by HPLC using a monolithic column

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Abstract-

An isocratic HPLC method for the determination of phenol and nitrophenols (4-nitrophenol, 2-nitrophenol, 4,6-dinitro-o-cresol and 2,4-dinitrophenol) has been developed and validated using 2-chlorophenol as internal standard (IS) and a monolithic column in tap water samples. Prior to HPLC, the method requires solid-phase extraction (SPE) using polymeric Lichrolut EN cartridges. The method development involved the study of methanol and acetonitrile as organic modifiers, pH and flow-rate using a Chromolith RP-18e (150 mm \times 4.6 mm I.D.) column. After comparing the performance of the separations obtained with both organic modifiers, the optimum separation of these compounds was achieved using 50 mM acetate buffer (pH 5.0)-acetonitrile (80:20, v/v) as mobile phase, 3 mL min⁻¹ flow-rate and UV detection at maximum absorbance wavelength. Under these conditions, all analytes were separated ($R_s \geq 2.0$) in an analysis time of less than 3.5 min and the most important validation parameters were evaluated. The recoveries obtained in the accuracy test for all phenols studied were in the 90–112% range using a preconcentration factor of 40, and the intraday and interday precisions [expressed as coefficient of variation (CV)] were smaller than 15%. Finally, the proposed method was applied to wastewater samples from several industries.

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