A new comprehensive method for simple phenols in wine, vinegar and spirit using SPE/UHPLC/high resolution tandem mass spectrometry.

Barnaba, C.[1], Nardin, T.[1], Malacarne, M.[1], Dellacassa, E.[2], Nicolini, G.[1] and Larcher, R.[1]

[1] *Centro Trasferimento Tecnologico, Fondazione E. Mach, via E. Mach 1, 38010 San Michele all'Adige (TN), Italia*. [2] *Universidad de la Republica Uruguay, Facultad de Quimica, Gral. Flores 2124, C.P. 11800, Montevideo, Uruguay. E-mail: roberto.larcher@fmach.it*

Natural phenolic compounds constitute a wide and complex group of plant secondary metabolites that are known for their great contribution to color and aroma of fruit and plant derivatives and for their anti-inflammatory, antioxidant, cardio protective and many other remarkable physiologic effects. Wine contains mainly phenolic acids, anthocyanins, tannins and flavonoids with significant differences between white and red wines.
The aim of the study was to develop a new comprehensive method to analyze the largest number of simple phenolic compounds in wine, vinegars, and distillates.
The sample was filtered, diluted 10 times and spiked with p-nitro phenol I.S.. SPE on-line (HyperSepTM Retain PEP spe cartridge), used to minimize matrix effects, was coupled with an ultra-HPLC (Thermo Ultimate R3000; Acquity UHPLC BEH C18 column) and a high resolution tandem mass spectrometry (Orbitrap Q-ExactiveTM; heated electrospray ionization in negative ion mode). Mass spectra were acquired in profile mode through a full MS-data dependent MS/MS analysis (full MS–dd MS/MS). Full mass spectra were recorded at mass resolving power of 140.000 full width at half-maximum (FWHM, calculated for m/z 200, 1.5 Hz) in the scan range of 50-750 m/z. Linearity (R2 > 0.99) was verify of 5 orders of magnitude for 50% of analytes, of 4 for 19.7%, of 3 orders for 17.8% and of 2 orders for 12.5%. Limits of quantitation were calculated according to Eurachem considering 12% R.S.D. as maximum acceptable value. Relative standard deviations were also used to define intra-day precision of method. For all analytes at concentration levels included in the linear range, R.S.D.% was less than 12% or better. Accuracy was evaluated from the recoveries of spiked samples having recoveries from 80 to 120% for 85% of compounds, 60 - 100% for 9%, and 105 to 133% for 6%. Inter-day method precision was evaluated achieving after 72 hours a relative standard deviation always less than 10%. This method proved to be adequate to analyze oenological matrices.

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