

Categoria do Resumo (Tahoma 11 – centralizado) Inserir a categoria do trabalho

**Multiresidue determination of pesticides in surface water by
SPE-HPLC-ESI and SPE-UPLC-ESI tandem mass spectrometry
(MS/MS – triple quadrupole analyzer)**

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The classical HPLC and UPLC (ultra performance liquid chromatography) methods, both combined with tandem mass spectrometry (MS/MS – triple quadrupole analyzer) and electrospray ionization (ESI) interface were used for the simultaneous determination of thirteen pesticides in surface water. The selected analytes sulfentrazone, picloram, 2,4-D, propanil, hexazinone, tebuthiuron, atrazine, ametryn, metribuzin, simazine, clomazone, molinate and diuron are the most used pesticides in sugarcane crops.

The analytes were extracted from 250 mL of sample (with adjusted pH at 2), by using solid-phase extraction (SPE). The cartridge used was Oasis[®] HLB and. Studies at fortification level of 0.2, 0.4 and 2 $\mu\text{g L}^{-1}$ gave mean recoveries ranging from 70 to 120 % for all compounds. The separation was carried out using an Acquity UPLC[®] BEH C18 column (1.7 μm , 2.1mm ID, 50mm) and Polaris C18 A column (5 μm , 2mm ID, 50mm) for UPLC and HPLC respectively and Mobile phase consisting of 0,1 % formic acid in water and methanol in gradient elution mode was carried out.

For the SPE-HPLC-ESI the analysis time was of 20 minutes while for SPE-UPLC-ESI the time was 3.5 minute. The limits of detection (LOD) for the ionized negatively pesticides (sulfentrazone, picloram, 2,4-D, propanil) were similar for both techniques. For the majority of the ionized positively compounds, the LODs were lower than 0.5 $\mu\text{g L}^{-1}$ for the SPE-UPLC-ESI.

The SPE-UPLC-ESI-MS/MS is powerful analytical technique for the rapid determination of pesticides at trace levels in environmental water.

Referências

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- [2] Sannino, A.; Bolzini, L.; Bandini, M. *Journal of Chromatography A*, **2004**, 1036, 161-169.