Determination of β-blockers in animal samples by different extraction methods.

β-blockers are used in animals, given by intramuscular injection, to calm them and to prevent death or even the loss of meat quality during transport to the slaughterhouse. Most tranquillizers are rapidly metabolised in the animal’s body. Any residues are concentrated in the kidney and/or liver. Eating food containing high levels of these substances can be harmful to consumer health, so the control of β-blockers residues is required.

In this context, the present work focused on the detection and quantification of four β-blockers in animal samples. Finally, this study shows which extraction method provides better recoveries, being faster and simpler.

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Determination of β-blockers in animal samples by different extraction methods followed by HPLC-UV and LC-MS analysis.

In this work, different analytical extraction methods (Membrane Assisted Solid Extraction “MASE”, Membrane Assisted Solid Extraction using Molecularly Imprinted Polymers “MASE-MIP”, Molecularly Imprinted Solid Phase Extraction “MISPE”) have been compared for the extraction of four β-blockers: atenolol, carazolol, metoprolol and propranolol in bovine meat and swine kidney. The procedure involves extraction with ethyl acetate. Extraction parameters such as the type of organic acceptor solvent, extraction time, addition of salt in the aqueous sample and amount of MIP were optimized.

All determination was performed applying high-performance liquid chromatography with UV detection at 260 nm. The chromatographic separation was performed in an Agilent Eclipse XDB-C18 column (150 mm x 4.6 mm, 5 μm) using a gradient mixture of (A) 10 mM ammonium formate in water (pH 3.9 adjusted with formic acid) and (B) methanol as mobile phase at a flow rate of 0.2 mL/min. LC-MS analysis was used to check the obtained results. The electrospray interface was operated in the positive ion mode. Resulting chromatograms were free of interfering peaks. The results are well below the current maximum residue limit for the carazolol compound.

In general, faster analysis and better extraction of β-blockers from animal tissues were achieved with MISPE. It is concluded that LC-MS is the more sensitive method than HPLC for this work.

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