ADVANCES IN THE SAMPLE PREPARATION.
GAS CHROMATOGRAPHIC IDENTIFICATION
AND QUANTIFICATION OF WATER SOLUBLE
POLLUTANTS

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An overview is presented on recent proposals and experience gained so far on sample
preparation and gas chromatographic identification/quantification of pollutants present in
the aquatic environment, with special regard to the Non-Steroid Anti-Inflammatory Drugs
(NSAIDs), including also other acidic drugs, estrogens, personal care products and
pollutants of industrial origin, as well as their metabolites. Published material was classified
and evaluated from an analytical viewpoint, with particular emphasis primarily on
pollutants’ enrichment procedures and secondly on Gas Chromatographic detection and
quantification techniques. Recently, more and more attention has been devoted worldwide
to the identification and quantification of pharmaceuticals, personal care products,
compounds of industrial origin and their metabolites (in the following referred to as
pollutants) present in the aquatic environment which can pose health risk to humans and
animals as well.

Since pollutants’ concentrations - in waste, riverine and drinking waters - can vary
between 10^-6 and <<10^-9 g/L, an enrichment step prior analysis is inescapable. This
enrichment step plays a crucial role in the reliable and reproducible determination of
pollutants. Among the major chromatographic methodologies, such as Gas
Chromatography (GC), High Performance Liquid Chromatography (HPLC) and Capillary
Electrophoresis (CE), GC and HPLC are those primarily applied, GC being often the
preferred option.

As HPLC techniques are complex and expensive, according to some research groups
standardization of the pharmaceutical residue analysis for regulatory purposes can be more
conveniently performed by coupling with Mass Spectrometry (MS) (GC-MS and/or GC-
MS/MS). In fact, in order to simultaneously identify and quantify as many as possible
differently functional-groups-containing compounds (as required for the
determination of water pollutants), GC separation combined with MS detection does
provide several advantages, i.e.: (i) high selectivity of MS and/or MS/MS detection
combined with chromatographic separation at the lowest cost; (ii) high detection power,
often equivalent to and in some cases higher than that offered by HPLC); (iii) outstanding
resolution of the GC capillary columns; (iv) availability and expediency of the silylation
approach in terms of proper labeling of all active-proton-containing functional groups in a
single step and of increase in the selectivity and sensitivity of the procedure. Last, but not
least, the reasonable cost of the GC-MS/MS systems compared to the corresponding
HPLC-MS/MS ones might be also attractive, thus paving the way to its acceptance in
specialistic laboratories.