

Elemental Speciation and its Application to RoHS and REACH Studies

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Balazs offers compliance analysis for several directives including RoHS (Restriction of Hazardous Substance) and REACH (Registration, Evaluation, Authorization and Restriction of Chemicals). RoHS studies which involve the quantitation of Cd, Hg, Pb, hexavalent chromium (CrVI), polybrominated biphenyls (PBB) and polybrominated biphenyl ethers (PBDE) are performed regularly in our laboratory. Sample preparation techniques involve homogenization, cryo-grinding and acid digestion for total element and water extraction or alkaline digestion for hexavalent chromium. Analysis techniques involve x-ray fluorescence (XRF) for elemental survey and inductively coupled plasma – mass spectrometry (ICP-MS) for total element content, colorimetry for CrVI and gas chromatography – mass spectrometry (GC-MS) for brominated compounds.

REACH is a more stringent regulation that lists “Substances of Very High Concern”... many of which are elemental species. Elemental species often vary in toxicity according to their chemical “forms” (e.g. oxidation state or compound group); therefore, methods must be developed to accurately determine the individual species. Speciation studies in our laboratory have been developed for arsenic pentoxide (AsV), arsenic trioxide (AsIII), sodium dichromate (CrVI), antimony trioxide (SbIII), antimony pentoxide (SbV), tributyltin (TBT) and triphenyltin (TPhT). The detection limits for these methods are in the sub to low ppb range, well below the REACH concentration limit requirements.

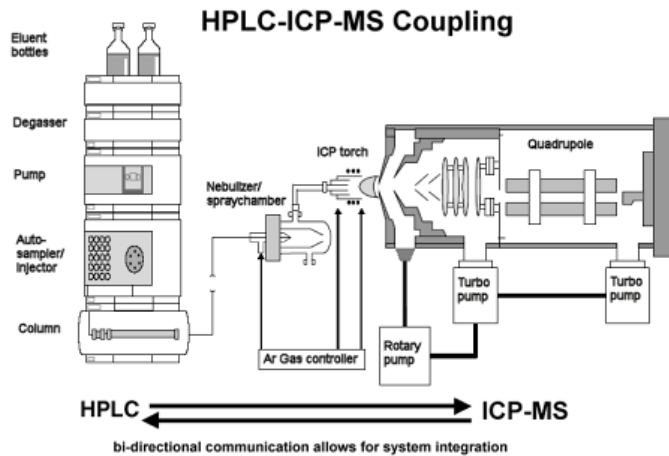
Typically for speciation analyses, a chromatographic method such as liquid or ion chromatography is coupled to an elemental detection method such as (ICP-MS). A sample preparation method that preserves the integrity of the individual element species often precedes the analysis.

Sample preparation methods used in our laboratory and pictured below include a Dionex accelerated solvent extractor (ASE) and a CEM MARS XPress microwave and vessels. The microwave was preferable for hexavalent chromium digestion as there was no contribution from the vessels unlike ASE which employs a stainless steel cell.



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The next couple of pictures show a schematic of the liquid chromatograph coupled to an ICP-MS and the interface of the instrumentation (Dionex GP50 IC with Agilent 7500s ICP-MS) in our laboratory, respectively.



A 10 ppb calibration standard chromatogram of AsIII, AsV, SbV and SbIII obtained using IC-ICPMS showing, all substance of very high concern, is shown below as well as a chromatogram of SbV and AsV which was present in a wafer sample subjected to ASE extraction followed by IC- ICPMS

