Selective Flow Injection Analysis of Ultra-trace Amounts of Cr(VI), Preconcentration of It by Solvent Extraction, and Determination by Electrothermal Atomic Absorption Spectrometry (ETAAS) - DTU Orbit (19/12/2018)

Selective Flow Injection Analysis of Ultra-trace Amounts of Cr(VI), Preconcentration of It by Solvent Extraction, and Determination by Electrothermal Atomic Absorption Spectrometry (ETAAS)

A rapid, robust, sensitive and selective time-based flow injection (FI) on-line solvent extraction system interfaced with electrothermal atomic absorption spectrometry (ETAAS) is described for analyzing ultra-trace amounts of Cr(VI). The sample is initially mixed on-line with isobutyl methyl ketone (IBMK). The Cr(VI) is complexed by reaction with ammonium pyrrolidine dithiocarbamate (APDC), and the non-charged Cr(VI)-PDC chelate formed is extracted into IBMK in a knotted reactor made from PTFE tubing. The organic extractant is separated from the aqueous phase by a gravity phase separator with a small conical cavity and delivered into a collector tube, from which 55 µl organic concentrate is subsequently introduced via an air flow into the graphite tube of the ETAAS instrument. The operations of the FI-system and the ETAAS detector are synchronously coupled. A significant advantage of the approach is that matrix constituents, such as high salt contents, effectively are eliminated. The extraction procedure was optimized by a simplex approach. A central composite design was subsequently employed to verify the estimated operational optimum. An 18-fold enhancement in sensitivity of Cr(VI) was achieved after preconcentration for 99 s at a sample flow rate of 5.5 ml min⁻¹, as compared to direct introduction of 55 µl of sample, yielding a detection limit (3σ) of 3.3 ng l⁻¹. The sampling frequency was 24.2 samples h⁻¹. The proposed method was successfully evaluated by analyzing a NIST Cr(VI)-reference material, synthetic seawater and waste waters, and waste water samples from an incineration plant and a desulphurization plant, respectively.

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