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Use of a microwave plasma torch coupled to electrochemical hydride generation for the optical emission spectrometric determination of As

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The use of a microwave plasma torch (MPT) with Ar as working gas coupled to an electrolysis cell was evaluated for the optical emission spectrometric (OES) determination of As. The microwave power, the plasma gas flow, the carrier gas flow, the electrolysis current, the concentration of H_2SO_4 in the catholyte and anolyte as well as the flow rate of the catholyte and the anolyte were optimized with respect to the net intensity for the As I 228.81 nm line and the precision. Under the optimized conditions a detection limit for As of 81 ng·mL^{-1} was obtained and the precision for the determination of As was found to be 2.0 % at the 3 µg·mL^{-1} level.

The interferences caused by the transition metals Cu(II), Fe(III) and Ni(II) in the range of 1 to 500 µg·mL^{-1}, the volatile hydride forming elements Sb(V), Se(VI) and Sn(IV) in the range of 1 to 100 µg·mL^{-1} as well as the depolariser NO_3^- in the range of 1 to 500 µg·mL^{-1} on the determination of As (3 µg·mL^{-1}) by EC-HG-MPT-OES were studied in detail. The presence of 100 µg·mL^{-1} Cu(II), Fe(II) and Ni(II) resulted in reduction of the signal for As by 96 %, 21 % and 57 %, respectively. The presence of 100 µg·mL^{-1} of the hydride forming elements Sb(V) and Sn(IV) caused an increase of the signal for As by 46 % and 76 %, respectively, whereas 100 µg·mL^{-1} of Se caused a suppression of 14 %. The concentration of HNO_3 was found to have no effect on the signal of As in the range investigated.

Also after an addition of the complexing agents L-cysteine and KI/ascorbic acid the interferences caused by the transition metals in the determination of As were investigated. It was found that in the presence of 100 µg·mL^{-1} of Cu and the addition of 2 % L-cysteine or KI/ascorbic acid (1:1), the relative net intensity for As was 26 % and 77 % as compared to 4 % in absence of the masking agents.

The influence of the electrolysis current, the concentration of the H_2SO_4 in the catholyte and the anolyte as well as the concentration of Cu(II) and Fe(III) in the catholyte on the amount of hydrogen produced in the cell was studied as well. The excitation temperature as measured with Ar I lines for different amounts of H_2 carried into the plasma was found to change from 4100 K without addition of H_2 to 5100 K for addition of 2 % H_2. The efficiency of the generation of AsH_3 was evaluated for different flow rates of the catholyte and the anolyte and for the electrolysis current. For this aim As was determined in the drain of the catholyte by FAAS.

The developed procedure was used for the determination of As in a coal fly ash (NIST SRM 1633a) and in two process water samples and the results of 1629 µg·mL^{-1} and 21.39 µg·mL^{-1}, respectively, were found to agree well with those attested or the values determined by ICP-OES. The procedure developed accordingly was found to be of good use when As is present as As(III), its suitability in the case of As(V) needs further investigation.