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Efficient generation of volatile cadmium species using Ti(III) and Ti(IV) and application to determination of cadmium by cold vapor generation inductively coupled plasma mass spectrometry (CVG-ICP-MS)

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A chemical vapor generation (CVG) method is developed for determination of cadmium (Cd) by ICP-MS. Titanium (III) and titanium (IV) were utilized for the first time as novel additives to enhance generation of volatile Cd species, and their synergistic effects were investigated for thiourea, L-cysteine and potassium cyanide (KCN) with different mineral acids. Both Ti(III) and Ti(IV) provided the highest enhancement with KCN. The improvement with thiourea was marginal (ca. 2-fold), while L-cysteine boosted signals slightly only with Ti(III) in H_2SO_4. Optimum CVG conditions were 4% HCl + 0.03 M Ti(III) + 0.16 M KCN and 2% HNO_3 + 0.03 M Ti(IV) + 0.16 M KCN. A 3% (m/v) $NaBH_4$ solution was adequate for successful vapor generation. Under these conditions, sensitivity was enhanced 40-fold with Ti(III) and 35-fold with Ti(IV), that are the highest enhancement factors achieved so far in Cd vapor generation. Detection limits (3s) were 3.2 and 6.4 ng L^{-1} for ^{111}Cd isotope using Ti(III) and Ti(IV), respectively. Evidence indicated that Ti(III) and Ti(IV) enhanced Cd vapor generation catalytically. Effects of transition metal ions, including Co(II), Cr(III), Cu(II), Fe(III), Mn(II), Ni(II) and Zn(II) were not significant up to 1.0 µg mL^{-1}. Among hydride forming elements, Bi, Pb, Sb and Sn depressed signals above 0.1 µg mL^{-1}. No interferences were observed from As(III) and Se(IV). The method was validated with determination of Cd by CVG-ICP-MS in certified reference materials, including Nearshore seawater (CASS-4), Bone ash (SRM 1400), Dogfish liver (DOLT-4), Mussel tissue (SRM 2976) and Domestic Sludge (SRM 2781).

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Development of new ion source/new desorption method for mass spectrometry

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Detection and quantification of illicit compounds at trace level is very much important for public health, security and safety. Mass spectrometry (MS) has already been demonstrated its versatility for detection and quantification of a wide range of compounds at trace level becauseMS can provide molecular level information of the target compounds. In MS, there are many ionization sources. So far, none of the ion source can ionize efficiently of a wide range of compounds. For example, electrospray ionization (ESI) and nano-ESI have been widely using for bio-molecules. Nano-ESI has shown better resolution than ESI but it has clogging problem. To overcome such a problem, several attempts have been taken to develop new ionization source, for example, MALDI, probe-ESI (PESI) etc. Recently we have developed new atmospheric pressure chemical ionization (APCI) using alternating current (ac) instead of dc and found as a soft ionization sourcefor explosives detection (*Habib et al.*, RCMS). Moreover, hollow cathode discharge (HCD) ion source has been fabricated for detection of explosives at trace level (*Habib et al.*, RCMS). A desorption method has also been developed using an ultra-cutter to desorb highly non-volatile illicit compounds and ionized dielectric discharge (DBD) ionsource,and found better limit of detection (LOD) (*Habib et al.*, ASMS). A vacuum glow discharge ionization (vacuum-GDI) source has been fabricated for the compounds those give negative ions. A further attempt has also been taken to fabricate a hybrid ionization source for polar/non-polar, volatile/non-volatile compounds.

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