Abstract

The aim of this thesis was to optimize the atomization conditions of volatile Ge species for hydride generation atomic absorption spectrometry (HG-AAS). The selected species were inorganic germanium (iGe) and its methyl-substituted forms: monomethylgermanium (MMGe) and dimethylgermanium (DMGe). The atomizers for which optimizations were performed included diffusion flame (DF), multi-atomizer (MMQTA), dielectric barrier discharge (DBD) either with sinusoidal modulation of the high voltage source and glued electrodes (REF-SIN) or sputtered electrodes with rectangular high voltage waveform modulation (SE-SW), and finally atmospheric pressure glow discharge (APGD).

Under optimal atomization conditions, the highest sensitivity, around 10 ms/ng, was found in DBD atomizers. The advantage of the SE-SW configuration, compared to the REF-SIN arrangement, is better peak shape and repeatability of the measurements. Atomizers such as DF and MMQTA provided mutually comparable sensitivity, which was about 5 times lower than in DBD. The APGD atomizer was the only atomizer studied in which the sensitivity was not comparavle among Ge species. The sensitivity for both methylated species was about half that for iGe (3.0 ms/ng).

As part of the mechanistic study, high Ge deposition rates in all types of atomizers, except DF, were demonstrated by leaching experiments, reaching 40-75%, indicating high reactivity and rapid decay of free atoms. The atomization of GeH_4 in DBD (SE-SW) and APGD atomizers was studied in more detail using laser-induced fluorescence (LIF) to visualize the spatial distribution of free atoms and to quantify the atomization efficiency. The efficiency reached 75% in APGD, while in DBD it does not exceed 5%. The formation of Ge deposits in the DF atomizer was studied using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The layer formed is not homogeneous and is probably composed of clusters of elemental Ge.

Key words:

atomic absorption spectrometry, germanium, monomethylgermanium, dimethylgermanium, hydride generation, diffusion flame (DF), multi-atomizer (MMQTA), dielectric barrier discharge (DBD), atmospheric pressure glow discharge (APGD), atomization of volatile compounds