SURFACE STUDIES IN SLOTTED QUARTZ TUBE ATOM TRAPPING FLAME ATOMIC ABSORPTION SPECTROMETRY FOR ANTIMONY DETERMINATION

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In order to improve the sensitivity of conventional flame atomic absorption spectrometry (FAAS), several atom trapping techniques have been introduced [1 review]. In slotted quartz tube atom trap flame atomic absorption spectrometry (SQT-AT-FAAS) system, a slotted silica tube positioned on the flame functions as a trap. The sample solution is aspirated for a fixed time and analyte species are collected on the inner surface of SQT. To atomize the collected species, flame conditions are changed by organic solvent aspiration.

The elements trapped on SQT have been shown to be in different states such as, the reduced state (metallic), in a partly reduced and partly oxidized state and, in the oxidized state [2, 3]. During revolatilization the removal process is not a simple reduction, direct contact with flame products is required; heating is not necessarily associated with the removal of surface species [4]. Further studies are required in order to have a clear picture of the trapping mechanism.

In this work, two identical quartz plates were placed inside an SQT during trapping and revolatilization stages. The quartz plates had dimensions of 10 x 85 x 1.0 mm. The plates were placed so that each one covers half of the SQT starting from the central position (0 mm) to one end (85 mm) inside. Four samplings were done, namely with no treatment, exposed to flame without any analyte aspiration, with flame using only trapping and finally with flame using both trapping and revolatilization. Raman and XRD techniques were used to investigate the quartz plate surfaces after each treatment. For Raman study the spectra were taken at 70 points on each sample. As a result of Raman and XRD studies, it was concluded that the amount of analyte species trapped is increasing vertically starting from the position of 6.0 mm until to reach a maximum value at 8.0 mm and decreasing afterwards. In horizontal scanning toward the ends of slotted quartz tube, the amount of analyte species trapped increases from the point of 10 mm to reach a maximum value at 25 mm and then decreasing to 40 mm. After revolatilization process, the surface returns almost to its original state as before trapping occurs. The data are indicative of the absence of any simple relationship between the surface temperature of silica and the trapping process. Examination of XRD data reveals that the species are trapped in the form of crystalline oxides; Sb$_2$O$_3$ and other oxides may be present.

KEYWORDS: Surface study, antimony, atom trapping, atomic absorption spectrometry.

REFERENCES: