A green and rapid method was developed for the simultaneous separation/preconcentration and sequential monitoring of arsenic, antimony and mercury by flow injection magnetic solid phase extraction coupled with on-line chemical vapor generation and determination by high resolution continuum source graphite furnace atomic absorption spectrometry. The system is based on chelating/cationic retention of the analytes onto a magnet based reactor designed to contain functionalized magnetic nanoparticles (MNPs). The MNP score allows overcoming the back-pressure problems that usually happen in SPME methods with NPs thanks to the possibility of immobilizing the MNPs by applying an external magnetic field. Several chemical and flow variables were considered as factors in the optimization process using central composite designs. With the optimized procedure the detection limits obtained were 0.2, 0.003 and 0.4 µg/L for As, Sb and Hg respectively. For the quality control of the analytical performance and the validation of the developed method the analysis of two certified samples TM 24.3 and TMDA 54.4 Fortified Lake Waters was addressed. The results showed good agreement with the certified values.