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**Separation-preconcentration of nickel and lead in food samples by  
a combination of solid–liquid–solid dispersive extraction using  
SiO<sub>2</sub> nanoparticles, ionic liquid-based dispersive liquid-liquid  
micro-extraction**

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**Abstract**

A microextraction method for the determination of nickel and lead using solid–liquid–solid dispersive extraction followed ionic liquid-based dispersive liquid-liquid microextraction (SLSDE-ILDLLME) was presented. It was applied to the extraction of nickel and lead from food samples. Ammonium pyrrolidine dithiocarbamate (APDC) as complexing agent, [C<sub>4</sub>MIM][PF<sub>6</sub>] as ionic liquid, SiO<sub>2</sub> as nanoparticles and 2 mol L<sup>-1</sup> HNO<sub>3</sub> as eluent were used. Several important parameters such as amount of IL, extraction time, pH and volume of the complexing agent were investigated. The quantitative recoveries were obtained at pH 7.0 for analytes. Under the optimum conditions, the limits of detection (LODs) calculated using  $3(Sd)_{blank}/m$  were 0.17 for Ni(II) and 0.79  $\mu\text{g L}^{-1}$  for Pb(II) for aqueous solutions with 125 enrichment factor (EF). The limit of detections of the analyte ions ( $3(Sd)_{blank}/m$ ) for solid samples were 0.09  $\mu\text{g g}^{-1}$  (Ni) and 0.40  $\mu\text{g g}^{-1}$  (Pb). The accuracy of proposed method was confirmed by the analysis of standard reference material (1577c bovine

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