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Abstract

Two operational modes for Lab-In-Syringe automation of direct-immersion single-drop microextraction have been developed and critically compared using lead in drinking water as the model analyte. Dithizone was used in the presence of masking additives as a sensitive chromogenic complexing reagent. The analytical procedure was carried out inside the void of an automatic syringe pump. Normal pump orientation was used to study extraction in a floating drop of a toluene-hexanol mixture. Placing the syringe upside-down allowed the use of a denser-than-water drop of chloroform for the extraction. A magnetic stirring bar was placed inside the syringe for homogenous mixing of the aqueous phase and enabled in-drop stirring in the second configuration while resulting in enhanced extraction efficiency. The use of a syringe as the extraction chamber allowed drop confinement and support by gravitational differences in the syringe inlet. Keeping the stirring rates low, problems related to solvent dispersion such as droplet collection were avoided. With a drop volume of 60 µL, limits of detection of 75 nmol L^{-1} and 23 nmol L^{-1} were achieved for the floating drop extraction and the in-drop stirring approaches, respectively. Both methods were characterized by repeatability with RSD typically below 5%, quantitative analyte recoveries, and analyte selectivity achieved by interference masking. Operational differences were critically compared. The proposed methods permitted the routine determination of lead in drinking water to be achieved in less than 6 min.