



**Method development for the recovery of trace metals from
unpreserved water samples**

By

Wenshuai Tan

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Certificate of Original Authorship

I hereby declare that this thesis has not been submitted, either in the same form or a different form, to UTS or any other university for the award of any other degree. To the best of my knowledge and belief, the thesis contains no material that has been previously published or written by another person except where references are made.

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Abstract

Water contamination by trace metals in natural and industrialised environments is a common phenomenon, the analysis of trace metals is therefore a very important process. Trace metal analysis is employed to assess the occurrence and concentration of contaminants thereby providing important data to protect our ecosystems and public health. During trace metal analysis, sample preservation is a critical requirement however it is also subject to numerous difficulties throughout the testing procedure.

The major standard water testing methods to preserve trace metals (as adopted by the USEPA and APHA) use nitric acid (HNO_3) which is added to water samples usually stored in polyethylene bottles. However, in some cases, samples are sent to laboratories unpreserved whereby significant amounts of trace metals may be lost due to being adsorbed in the inner walls of the plastic bottles, leading to a loss of metal concentration in the water samples. The analytical results of analysis could therefore be significantly lower than their true values.

To provide a solution to this problem, this study is focused on the recovery of 19 trace metals – Al, Ag, As, Ba, B, Cd, Cr, Co, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Tl, V and Zn – from unpreserved water samples spiked with trace metals.

First the thesis focuses on deionised (DI) water samples spiked with trace metals. Findings from this work with DI water samples have indicated that sample testing with 1% (v/v) nitric acid plus 0.01% (v/v) hydrochloric acid (HCl) for a 24-hour period enabled efficient re-extraction of 70-110% of trace metals from the tested water samples. However, mercury (Hg) did not yield high recovery results (around 56%) with this method although, with the addition of 10 mg/L Au solution, recovery of Hg was increased to about 70%.

After DI water samples recovery testing, then the thesis focuses on the recovery of trace metals from unpreserved drinking water samples. Tap water samples were studied with

this research method: 1% (v/v) nitric acid plus 1% (v/v) hydrochloric acid for a 24-hour period. This enabled efficient re-extraction of 70-110% of trace metals from the water sample. However, as with tap water sampling, mercury did not yield high recovery results with this method (around 66%) although, with the addition of 1 mg/L Au solution, the recovery of Hg was increased to about 78%. Therefore, it is highly recommended that acidified water samples – those containing 1% (v/v) nitric acid plus 0.01% (v/v) hydrochloric acid – are sent to the laboratory for testing whenever possible to guarantee greatest accuracy in trace metals analyses. If unpreserved tap water samples are received, 1% (v/v) nitric acid plus 1% (v/v) hydrochloric acid can be utilised to recover most trace metals, while 1 mg/L Au solution guarantees greatest accuracy when testing for mercury. Finally, the limitations of this study have been stated with recommendations for future studies and applications which might develop methods for the recovery of trace metals from unpreserved water samples.