# Determination of Trace Level Lead (Pb) in Drinking Water Using a Mercury-Free Electrode and a Portable Instrument According to the Requirements of USEPA Lead and Copper Rule

J. GANDHI[1] AND B. ZUMBRÄGEL[2]

## INTRODUCTION

Lead is known to be highly toxic, and lead salts are easily resorbed by humans. Cases of chronic lead poisoning caused by lead metal used in the water piping system are well known. Therefore, the control of drinking water for lead content is of utmost importance. The Lead and Copper Rule (LCR) published by the USEPA (United States Environmental Protection Agency) states an action limit of 15 µg/L lead for drinking water [**1**]. Using a portable voltammetric instrument, lead can be determined in these concentrations directly at the point of sampling.

## EXPERIMENTAL

### PRINCIPLE

The determination of lead in drinking water can be carried out by anodic stripping voltammetry (ASV) [**2,3**]. During the deposition step, lead ions are reduced and deposited on the gold working electrode which is modified with a silver film. During the stripping step, the deposited lead is stripped off allowing determination down to 2 µg/L Pb with a deposition time of 30 s. Lower concentrations can be quantified by increasing the deposition time. The concentration is calculated using the standard addition technique.

### INSTRUMENT AND ELECTRODE

The 946 Portable VA Analyzer is a battery-powered voltammetric analyzer which is intended for on-site analysis. The electrode used in this setup is the scTRACE Gold a combined voltammetric sensor with a gold micro-wire working electrode. The Ag/AgCl reference electrode and the carbon auxiliary electrode are located on the back of the sensor. For the determination of trace lead levels, the gold micro-wire is modified with a silver film. The usual use of mercury for the voltammtric determination of lead is not necessary. This application is completely mercury-free.

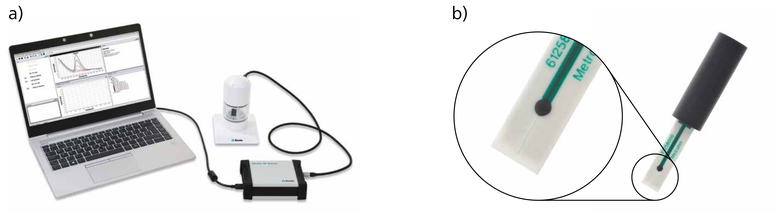
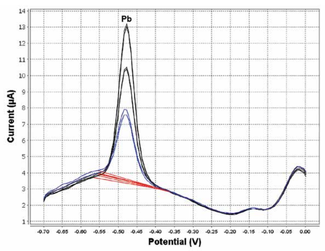



Fig. 1: a) 946 Portable VA Analyzer b) front side of the scTRACE Gold electrode, enlarged gold micro-wire working electrode.

## RESULTS



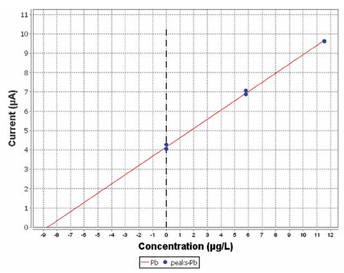




Fig. 2: Example for the determination of Pb in tap water (sample spiked with 15 µg/L Pb) with the 946 Portable VA Analyzer, result 14.8 µg/L Pb (recovery 98.7%).

### METHOD DETECTION LIMIT (MDL)

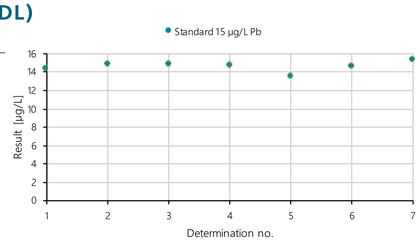



Fig. 3: Results of the laboratory fortified blank (β(Pb) = 15 µg/L) used for the determination of the MDL.

Method detection limit (MDL) = (t · S) = 3.143 · 0.55 µg/L = 1.72 µg/L
With: t – factor Student's t-distribution for 6 degrees of freedom, S – standard deviation
Minimum level (ML) = 3.18 · MDL = 3.18 · 1.72 µg/L = 5.47 µg/L

### ACCURACY AND PRECISION

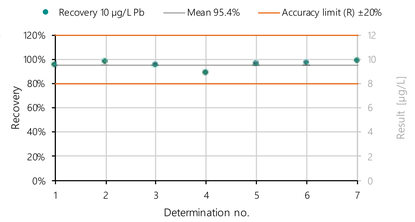



Fig. 4: Results for standard with β(Pb) = 10 µg/L for initial demonstration of accuracy and precision.

| | Limit [4] | Results | |
|---|---|---|---|
| Initial demonstration of capability Accuracy (as % recovery R) | 80%–120% | 88.6%–98.5% | Mean 95.4% |
| Initial demonstration of capability Precision (as % Relative Standard Deviation *s*) | ±20% | ±3.4% | |

### QUALITY CONTROL STANDARD

| Parameter | Certified Value | Uncertainty | QC Performance Acceptance Limit | Result | Recovery |
|---|---|---|---|---|---|
| Lead | 63.6 µg/L | 2.99% | 55.3–71.9 µg/L | 66.9 µg/L | 105.1% |

### RECOVERY FORTIFIED SAMPLES

**Samples:**
- Mineral water with high calcium and sulfate content
- Tap water

Both samples: Pb < MDL

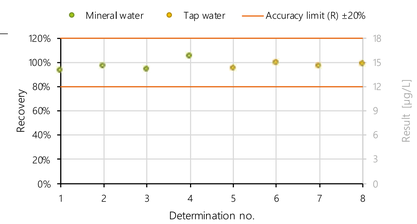



Fig. 5: Recovery of lead in mineral water and tap water (samples fortified with β(Pb) = 15 µg/L).

All statistical data were obtained following the recommendation in literature [**5**].

## CONCLUSIONS

- Being completly mercury-free, this application is environmentally friendly.
- With the calculated method detection limit (MDL) of β(Pb) = 2 µg/L and the resulting minimum level (ML) of β(Pb) = 5 µg/L, the method is sensitive enough to reliably monitor the action limit of 15 µg/L Pb as specified in the USEPA Lead and Copper Rule.
- The accuracy and precision of the method fulfills the quality control targets used by the Office of Ground Water and Drinking Water.
- The accuracy found in standard solutions can equally be applied to drinking water samples.
- Using the 946 Portable VA Analyzer, the determination of trace concentration of lead in drinking water can be carried out independently from a laboratory.

1 **Metrohm USA, jayesh.gandhi@metrohmusa.com**

2 **Metrohm International Headquarters, Switzerland, barbara.zumbraegel@metrohm.com**

8.000.6113EN

**REFERENCES**

[1] Lead and Copper Rule, United States Environmental Protection Agency, https://www.epa.gov/ground-water-and-drinking-water/revised-lead-and-copper-rule

[2] Metrohm Application Bulletin 433/1 – Determination of lead in water with the scTRACE Gold modified with a silver film

[3] Metrohm Application Work AW VA CH4-0587-092019 – Determination of Pb in acidified water samples at a silver film electrode using the 946 Portable VA Analyzer

[4] Protocol for the Evaluation of Alternate Test Procedures for Organic and Inorganic Analytes in Drinking Water (EPA 815-R-15-007), February 2015

[5] Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater Under EPA's Alternate Test Procedure Program (EPA 821-B-18-001), February 2018

Metrohm