

References

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Iridium Microelectrode for Testing Water Quality

The lack of sound environmental management of our natural resources is of growing concern, and increasingly legislation is setting standards for the pollutants which can be released into the environment. Pollution in river water, however, is now routinely checked, and improving the quality of the water into which industrial and domestic effluent may be discharged is one aim of the national River Authorities.

Water is tested for the presence and quantity of many different types of pollutants, including organic pollutants, such as pesticides and insecticides, nitrates and nitrites, and the elements, such as the heavy metals: lead, cadmium and copper. A number of analytical techniques are presently used to measure the amounts of around 6000 individual pollutants, mainly by laboratory analysis from collected samples.

However, an advance on the technique for monitoring lead and cadmium using an improved mercury-based iridium microelectrode is suggested in a paper from the University of Geneva (M.-L. Tercier, N. Parthasarathy and J. Buffle, *Electroanalysis*, 1995, 7, (1), 55-63).

The drawbacks to using microelectrodes for water analysis have been a lack of reproducibility in their fabrication and the difficulty in characterising them, and since complete reliability is necessary in the analysis of water contaminants, microelectrodes have not been used. However, based on prior research on ultramicroelectrodes, the researchers have produced a sturdily constructed, reliable mercury-plated iridium-based microelectrode, having a central iridium electrode of radius 62 μm , which gives reproducible results. This has been achieved by thorough attention to all aspects of the fabrication process, in particular, since the electrode is built within a glass pipette, to the iridium-glass sealing, and to the iridium-copper soldering in order to obtain perfect electrical contact. The morphology of the iridium disk is very important and strongly depends on using a reproducible standard polishing procedure.

Optimal polishing procedures and conditions are given to achieve this. The build up of electrostatic charge on the tip of the microelectrode is avoided by shielding the glass pipette with an outer layer of palladium.

To date the system has achieved near 100 per cent reproducibility and reliability for the measurement of trace metals by differential pulse anodic stripping voltammetry and square wave anodic stripping voltammetry (SWASV). Electrodes have been used continuously for several days without requiring a renewal of the mercury layer.

When tested using SWASV in river water, which contained a high level of suspended particle content, trace amounts of free cadmium and lead were able to be measured in concentrations as low as 0.5 nM and 0.1 nM, respectively. Direct SWASV measurements using such a microelectrode were the best for routine analysis of dissolved trace metals in fresh water, since problems caused by contamination and/or adsorption are minimised. This method allowed the free and total metal concentrations to be rapidly determined. These are the most important trace metal speciation parameters. Sample pretreatment is not required. At present the lifetime of these state-of-the-art microelectrodes is more than 2 years.

If dissolved organic matter in low concentrations of 1 to 2 mg/litre, relative to particle load, is present then trace metals at levels of 50 pm can be detected, although high levels of particles do not hinder SWASV measurements. To avoid problems in water with higher dissolved organic content and to extend the use of the sensor for *in situ* measurements of any type of natural water, a membrane coated microsensor is being developed. Other uses suggested for this sensor include an *in situ* automatic quality control system for industrial application, the measurements of samples of small volume, being used in still waters and as part of other routine detection systems.