

PS ANALYTICAL

Application Note 004

TOTAL MERCURY DETERMINATIONS IN INDUSTRIAL AND DOMESTIC WASTE WATERS

Introduction

Mercury determinations in waters represent a significant part of many routine laboratories' workloads. The instrumentation and method used must provide quick, accurate and sensitive results. Sample preparation by bromination and detection using the Merlin Plus system fulfil all these requirements and more.

Procedures

Adding potassium bromate/bromide and hydrochloric acid and heating for 4 hours before analysis prepare samples. There must be an excess of bromine at all times. Blank solutions should be prepared using the same reagents. Samples may be brominated at the time of collection. Once treated, samples have been found to be stable for at least five days. Tin (II) chloride is used as the reducing agent for continuous flow vapour generation and should be prepared daily.

Typical instrument values for the vapour generator are as follows: **Delay time - 15 secs; Rise time - 25 secs; Analysis time - 30 secs; Memory time - 60 secs.**

The Merlin detector should be calibrated to the concentration range expected in the samples. For a 0 - 1 ppb calibration range the Merlin is normally run at a range of 2000. The maximum range is 10,000 which shows the sensitivity available.

Results

Figure 1 shows a typical calibration curve for 0 - 1 ppb, and Figure 2 a peak shape from a 0.2 ppb standard. The system has a **Method Detection Limit of below 5 ppt ($3\sigma_{n-1}$) assuming a 5 times dilution of the sample for water analysis.** The linear dynamic range is between 1 and 100 ppb when using continuous flow and can be extended to 10 ppm using discrete sample injection.

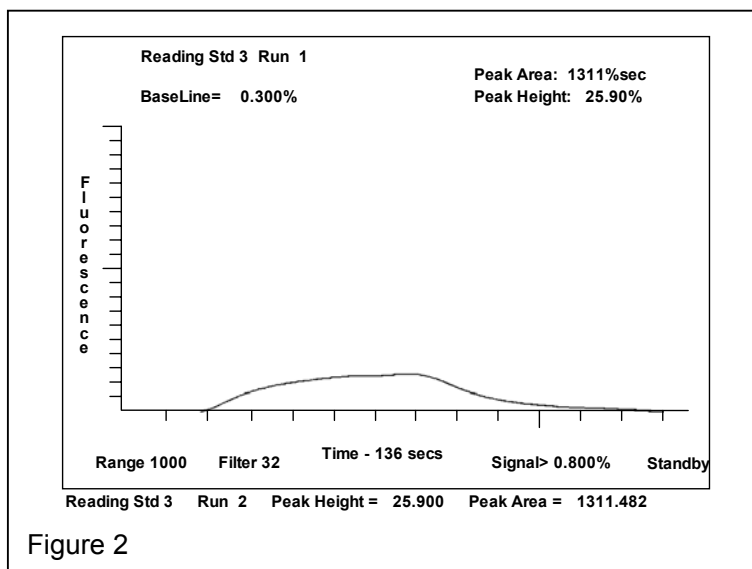
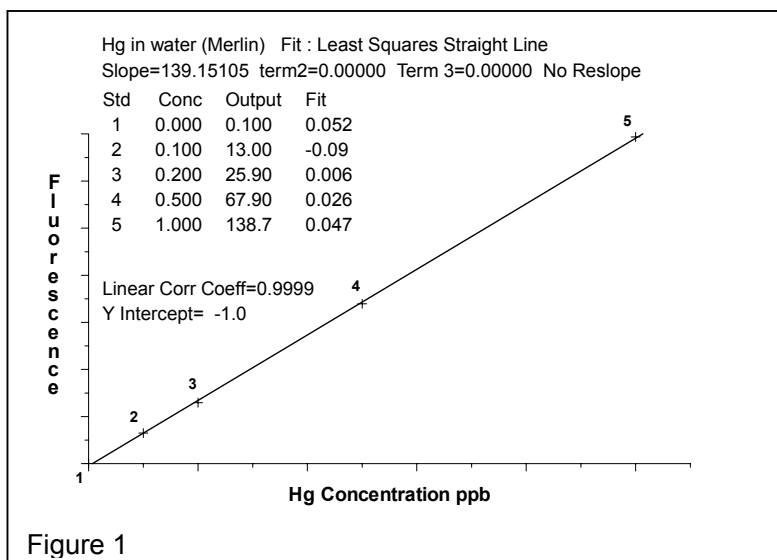


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Conclusion

The bromination digestion procedure overcomes most interference such as the formation of mercury complexes with organic materials, and will oxidise sulphide. Chloride interferences are also overcome; therefore the procedure is suitable for analysing saline waters. Compared to other digestion procedures bromination is extremely rapid, and because it.



occurs at room temperature mercury is not lost through vaporisation. All the reagents can be obtained at extremely high purity, thus minimising background levels. **The combination of bromination with vapour generation and atomic fluorescence detection allows excellent sensitivity, accuracy and ease of use.** Peak shapes are a useful diagnostic tool; regular shapes indicate good chemistry and no interferences. In conjunction with a PSA 20.100 random access autosampler and PSA's Control and Data Management Software up to 40

samples per hour can be analysed. When using the 10.004 vapour generator as a discrete injection valve up to 80 samples per hour can be analysed.

Please contact P S Analytical for a full method describing this procedure.

Please Note: This Application Note was carried out using the 10.623 Merlin Plus system. This has now been superseded by the Millennium Merlin, which has 10x greater sensitivity and a wider linear range.



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