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Temperature Effects in Mercury Analysis

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Temperature Effects in Mercury Analysis

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Flameless atomic absorption determination of mercury involves blowing elemental mercury from reducing solution into a spectrophotometer cell. The temperature of the reducing solution greatly affects the rate at which mercury comes out of solution, and thus, if not kept constant, can affect analytical results.

KEY WORDS: Mercury, flameless atomic absorption, temperature effects.

Flameless atomic absorption or fluorescence spectrophotometry is the most usual final step in the determination of mercury in environmental samples. Hg^{II} in solution is reduced by SnCl_2 to Hg^0 , and the mercury blown out of solution as vapour into a spectrophotometer cell. The absorbance¹ or fluorescence² of the vapour is measured, giving a characteristically shaped recorder trace. Figure 1 shows the peaks obtained with an open fluorescence system—i.e. the mercury vapour is not recycled through the cell. The heights of the peaks for standards are measured and a calibration curve obtained.

Peak height depends on a number of factors—total mercury in the reducing solution, cell size, dead volume in the aeration system, and the rate of release of mercury vapour from the solution. Factors that affect this rate of release are solution volume³, gas flow rate³ and acid normality.⁴ All these are well documented. However, it is often not appreciated that solution temperature also affects release rate. Radiochemical studies here have shown that the rate of release of mercury vapour can be doubled by an increase in solution temperature from 15°C to 40°C. This gives an approximate doubling of the recorder response with the particular instrumentation and aeration system

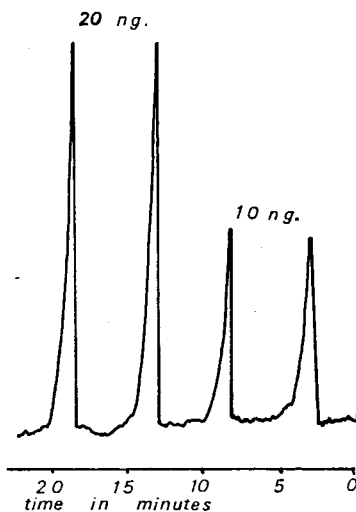


FIGURE 1 Atomic fluorescence spectrophotometry—typical recorder trace for 10 and 20 nanogram mercury standards.

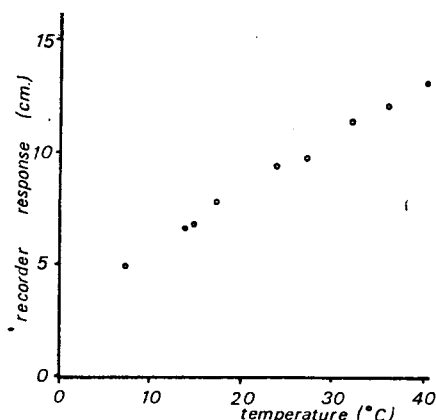


FIGURE 2 Relationship between solution temperature and the recorder response to a standard amount (30 nanograms) of mercury.

used here, in which an Evans Electroselenium 240 atomic absorption spectrophotometer is fitted with a modified Shandon Southern mercury fluorescence unit. Figure 2 shows a typical relationship between solution temperature and recorder response for a standard amount of mercury.

It is worth noting that bubbling gas through a solution will reduce its temperature. Thus the addition of a standard to a previously aerated solution⁵ to obtain a calibration may give misleading results.

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