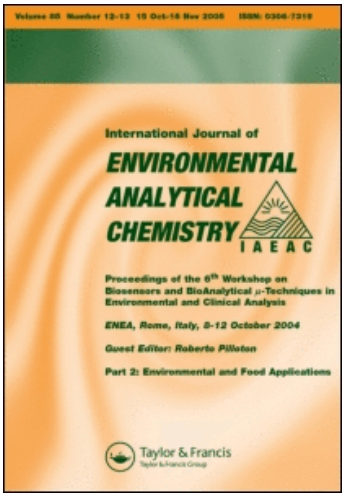

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

Determination of Selenium in Tissues, Serum, and Blood of Wild Rodents by Graphite Furnace Atomic Absorption Spectrophotometry

Chester D. Rail[a]; David E. Kidd[b]; William M. Hadley[c]
[a] Department of Environmental Health, Research, and Development, Albuquerque, New Mexico, USA [b] Department of Biology, University of New Mexico, Albuquerque, New Mexico, USA [c] College of Pharmacy University of New Mexico, Albuquerque, New Mexico, USA

To cite this Article Rail, Chester D. , Kidd, David E. and Hadley, William M.(1980) 'Determination of Selenium in Tissues, Serum, and Blood of Wild Rodents by Graphite Furnace Atomic Absorption Spectrophotometry', International Journal of Environmental Analytical Chemistry, 8: 2, 79 — 87
To link to this Article: DOI: 10.1080/03067318008071884
URL: http://dx.doi.org/10.1080/03067318008071884

Intern. J. Environ. Anal. Chem., 1980, Vol. 8, pp. 79–87
0306-7319/80/0802-0079 $6.50/0

Printed in Great Britain

Determination of Selenium in Tissues, Serum, and Blood of Wild Rodents by Graphite Furnace Atomic Absorption Spectrophotometry

CHESTER D. RAIL

Department of Environmental Health, Research, and Development, P.O. Box 1293, Albuquerque, New Mexico 87103, USA

DAVID E. KIDD

Department of Biology, University of New Mexico, Albuquerque, New Mexico 87131, USA

WILLIAM M. HADLEY

College of Pharmacy, University of New Mexico, Albuquerque, New Mexico 87131, USA

(*Received November 30, 1979; in final form January 7, 1980*)

Selenium (Se) levels were determined in liver, kidney, blood, and serum of wild rodents using graphite furnace atomic absorption spectrophotometry. A simple wet digestion technique was used to prepare samples for analysis. Nickel nitrate was used to suppress volatilization of Se during the char stage. Compared to existing techniques this greatly reduces the effort and time required for sample preparation and allows use of the graphite furnace atomic absorption technique for Se determinations.

KEY WORDS: Selenium, atomic absorption, tissues, blood, serum, graphite furnace.

INTRODUCTION

The increasing interest in determining Selenium (Se) levels in biological samples accentuates the need for a rapid, sensitive, economical, and reliable analytical procedure for the element.[1–5]

Se analysis by atomic absorption spectrophotometry (Argon-Hydrogen Generation System) is a reliable method, but it is time-consuming, expensive, and requires specialized equipment and personnel.[6–9]

An economical digestion procedure and a more rapid analysis process has been developed for Se in small amounts of tissue, whole blood, and serum. The purpose of this article is to report on the method developed.

METHODS

A Perkin-Elmer 306 atomic absorption spectrophotometer equipped with a Perkin-Elmer Model HGA-2100 graphite furnace, a Deuterium Background Corrector, and a Perkin-Elmer Se Electrodeless Discharge Lamp (EDL) was used for the analysis. A Houston Instruments Omniscribe recorder set on a 1 millivolt span recorded peak signals. The Se EDL was operated at 6 watts, wavelength setting of 196.0 nm, and a spectral slit width of 0.7 nm.

The HGA graphite furnace was programmed for drying at 125°C (50 sec), charring at 550°C (10 sec) and atomization at 2500–2700°C (4–5 sec). Argon was the furnace purge gas at a flow rate of 2 liters/minute. The flow rate was interrupted automatically during atomization. Samples were injected into the furnace by use of microliter pipettes (10–50 ul) with disposable tips.

Reagents and standards

1. Perchloric Acid (70%), ACS reagent grade.
2. Hydrogen Peroxide (30%), reagent grade.
3. Nickel Nitrate (1%). Dissolve 5 g of ACS reagent grade in 100 ml of doubly distilled water.
4. NBS Bovine Liver (1577), National Bureau of Standards Reference Material.

Sample preparation and procedure

Liver and kidney samples (entire tissues) were homogenized using a Braun-Sonic 1510 ultrasonic system (2 min/sample). Tissues were dried to constant weight in a drying oven at 60°C for 72 hours. Aliquots of tissue (0.05–0.25 g dry wt) were then placed into 15 ml (or larger) acid washed blood vial tubes with plastic screw caps. A digestion mixture of 70% perchloric acid and 30% hydrogen peroxide was added to each vial. The mixture was prepared in the ratio of 0.5 ml perchloric acid to 1.0 ml hydrogen peroxide per 0.1 g dry wt tissue sample or 0.1 ml blood or serum sample.

The digestion mixture was placed in a 70°C water bath for 6–7 hours or

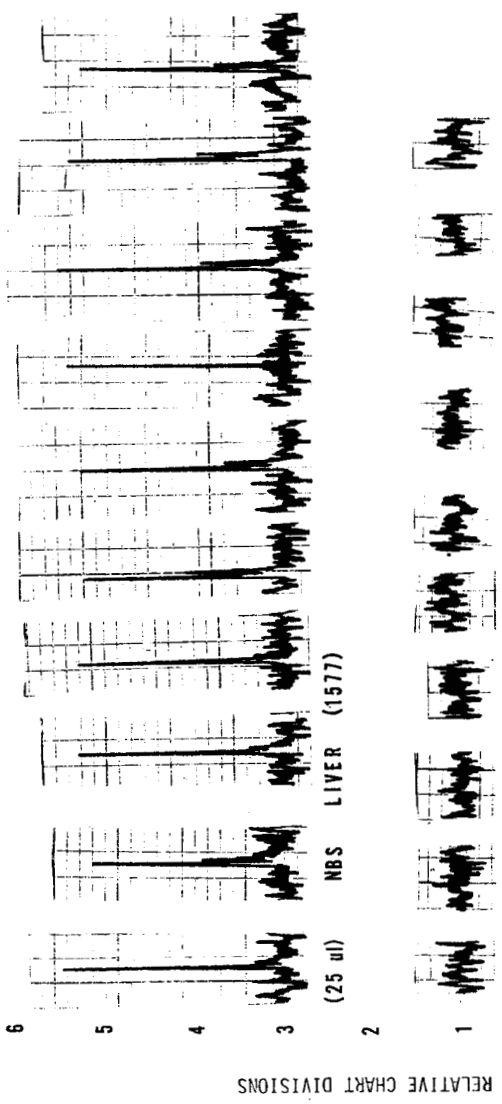

FIGURE 1 Ten replicate measurements of a (25 ul) injection of a tissue standard (NBS, 1577) and a blank solution into the HGA-2100.

until the sample had cleared. After digestion, 7.5 ml of doubly distilled water and 1.0 ml of the nickel nitrate solution were then added. This brought the total volume to 10 ml.

The procedure for processing blood and serum involved the same ratio of perchloric acid to hydrogen peroxide. If 0.1 ml of blood or serum was added, then 0.5 ml of perchloric and 1.0 ml of hydrogen peroxide were used. After digestion (same procedure as for tissues) 7.5 ml of doubly distilled water and 1.0 ml of nickel nitrate solution were added.

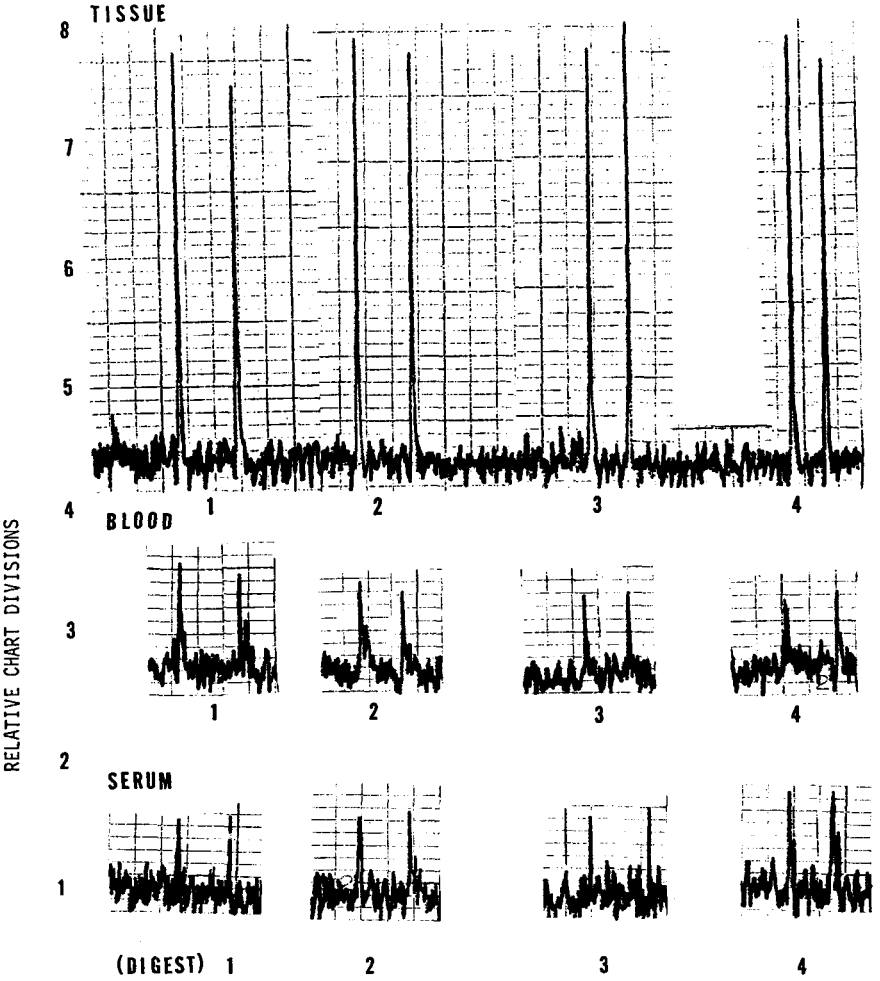

FIGURE 2 Reproducibility of the determination of Se in wild rodent tissue, blood, and serum; duplicate measurements.

In some instances up to 0.25 g dry wt of tissue and/or 0.2 ml of blood or serum could be digested by the 0.5 ml perchloric acid and 1.0 ml hydrogen peroxide additions.

NBS Standard Tissue Material (1577), blanks, and aqueous standards were prepared in the same manner.

The samples were now ready for analysis in the atomic absorption spectrophotometer.

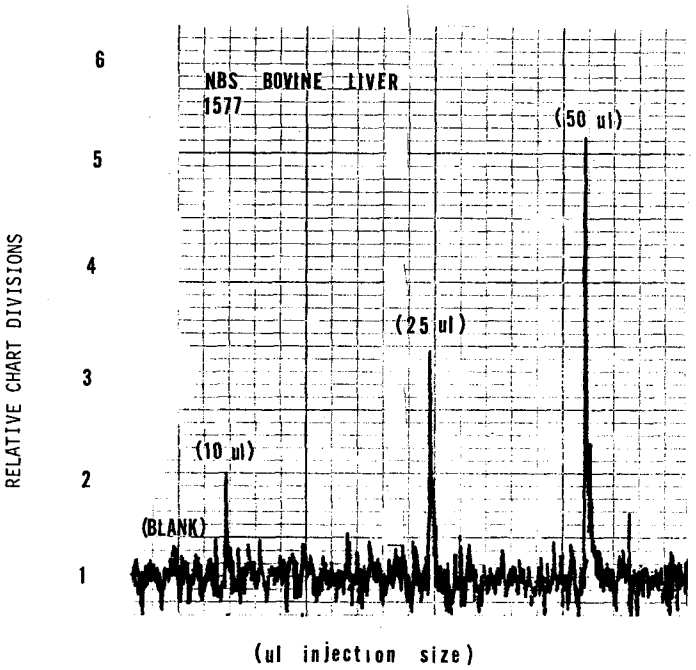

FIGURE 3 Recorder tracing for determination of Se in liver tissue (NBS, 1577); duplicate measurements.

RESULTS

Precision and detection limits

Ten replicate determinations of the Se peak by graphite furnace atomic absorption spectrophotometry (25 ul of digest of NBS Standard Reference Material) are shown in Figure 1. The average peak signal was 4.30 CM with a standard deviation of 0.22 CM. A similar precision was obtained for replicate determination of the Se peak in digests (25 ul) of wild rodent tissue, blood, and serum (Figure 2).

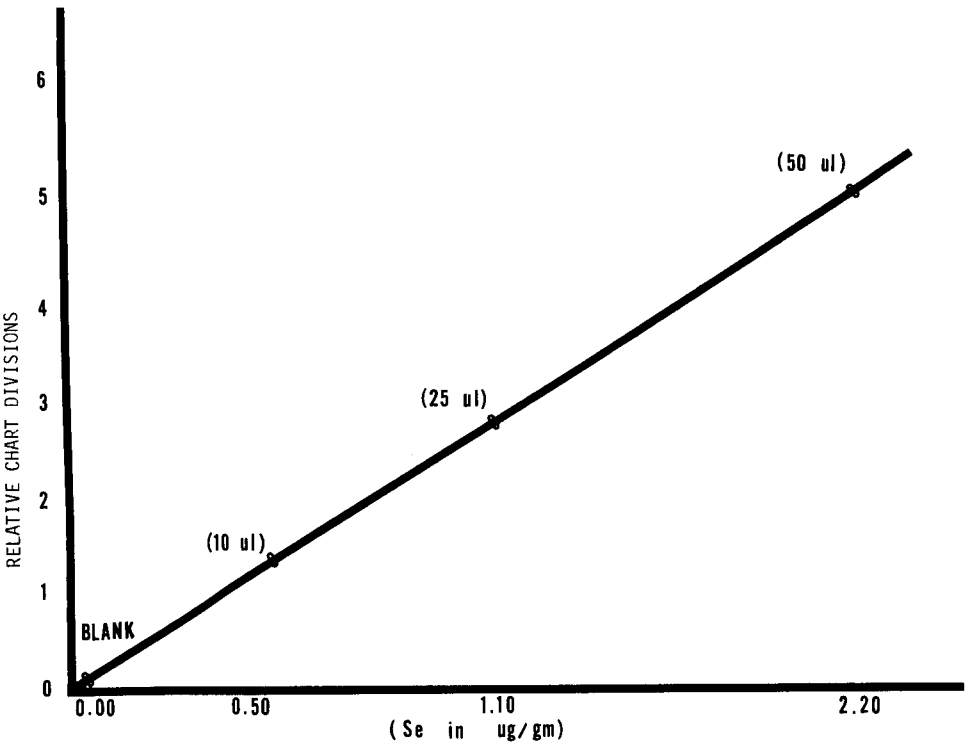

FIGURE 4 Calibration curve for Se standard tissue (NBS, 1577); duplicate measurements.

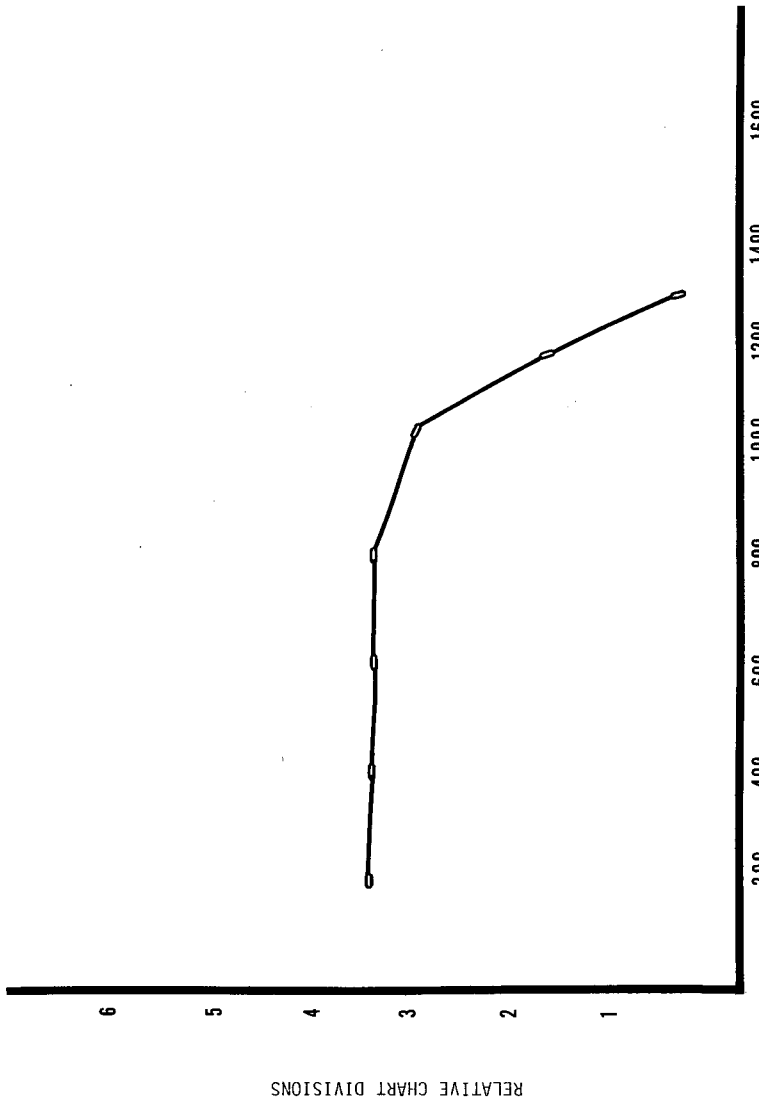

FIGURE 5 Effect of charring temperature on the atomizing signal (NBS, 1577); duplicate measurements.

For Se, the level of detection was 0.05 ug/g where the limit of detection was the level that gives a signal equal to twice the background noise. A typical recorder tracing of a calibration curve (NBS, 1577) with the use of 10–50 ul pipettes is shown in Figure 3 (25 ul = 1.1 ug/g). The curve is linear over the entire range (Figure 4).

Figure 5 shows the effects of charring temperature on atomizing signal response for the NBS Standard tissue sample. The charring temperature of 550°C was used for analysis of samples so as to increase the longevity of the graphite tubes.

Per cent recovery tests showed that Aqueous Standards were at a level of 120% compared to the Method of Additions which gave a % recovery of 80–100%.

DISCUSSION

Rapid and economical analysis of large numbers of small tissue samples for Se is possible using the reported method. The wet digestion method is economical and rapid. It eliminates the potential explosive hazard associated with the use of full strength perchloric acid in tissue digestion. The use of nickel nitrate to prevent the volatilization of Se, although the samples analyzed were primarily water and sludge materials, has been reported by several investigators.[4,5,7]

The digestion and analytical process has been used in the laboratory for analysis of Se in small samples of tissues (0.05–0.25 g dry wt), blood (0.1–0.25 ml), and serum (0.1–0.25 ml) from wild rodents. Many of the previous analytical methods for Se in tissues, blood, and serum have required large amounts of sample (1 g, 1 ml). The small sample size required allows Se analysis of tissues, blood, and serum from individual animals.

The estimated cost saving of this method of Se analysis is 10-fold over the Argon-Hydrogen Generation Method and 10-fold over other wet digestive techniques. The major expense is for graphite rods (approximately 100 analyses per rod). These efficiencies should make this an attractive preferred procedure for laboratories involved in routine analysis of many samples.

References

1. A. R. Zingaro and W. C. Cooper, *Selenium.* Van Rostrand Reinhold Co. New York (1974).
2. J. H. Watkinson, Symposium: *Selenium in Biomedicine*, Chap. 6, O. H. Muth, ed., Westport Ct. AVI Publishing Co., Inc. (1967).
3. D. Frost and P. Lish, *Ann. Rev. Phar.* **15,** 259 (1975).
4. C. D. Rail and W. M. Hadley, *J. Env. Health.* **39,** 173 (1976).

5. O. E. Olsen, *Methods of Biochem. Ana.* **21.** David Glick, ed. p. 39–78. (1973).
6. K. G. Brodie, *American Laboratory* **9,** 76 (1977).
7. D. T. Mahin and R. T. Lofberg, *Anal. Biochem.* **16,** 500 (1966).
8. R. F. Ediger, *At. Absorpt. Newsl.* **14,** 127 (1975).
9. T. D. Martin, J. F. Kopp and R. D. Ediger, *At. Absorpt. Newsl.* **14,** 109 (1975).