Analysis of Trace Elements in Animal Tissues

III. Determination of Manganese by Graphite Furnace Atomic Absorption Spectrophotometry

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Abstract

Graphite furnace atomic absorption spectrophotometry is a method used for the measurement of low concentrations of manganese (ppb range). Despite the widespread use of this technique, there is considerable inconsistency concerning sample preparation and choice of instrumental parameters. In this paper, we determined manganese concentrations of National Bureau of Standards (NBS) bovine liver by both graphite furnace (Instrumentation Laboratory IL 555B) and flame atomic absorption following wet digestion of the sample with nitric acid. The following instrumental parameters for the graphite furnace were found optimal for the measurement of manganese in digested NBS bovine liver: inert gas flow = 14 SCFH, drying temperature 100°C/15 s (step 1), 125°C/15 s (step 2), pyrolysis temperature 500°C/15 s (step 3), and 1000°C/15 s (step 4); atomization temperature 2250°C/10 s (step 5). For optimal results, the nitric acid concentration of the sample should be between 2 and 4M. There were no significant differences found for manganese concentrations determined by either peak height or peak area measurement. Additionally, no significant differences were found in manganese concentrations determined by flame or furnace methods. Assuming proper sample preparation and choice of instrumental parameters, values obtained for manganese concentration by graphite furnace and flame atomic absorption spectrophotometry are similar. Therefore, data obtained by these two methods can be compared directly.
Index Entries: Manganese determination, by graphite furnace AAS; atomic absorption spectrophotometry, of Mn in animal tissues; biological samples; biotrace element analysis, of Mn; analysis of biological trace elements; instrumental parameters, in biological trace element analysis; methods in biological trace element analysis.

Introduction

The essentiality of the trace element manganese for animals was established in 1931, but over 50 years later its metabolism is still poorly understood. A major reason for this lack of information has been the difficulty of its measurement; manganese occurs in very low levels in animal tissues, with a concentration range of 0.001 to 5 μg/g wet wt. These low concentrations necessitate the use of very sensitive analytical methods.

Neutron activation analysis and X-ray fluorescence may have the desired sensitivity and have been used to measure manganese (1, 2), but these methods are cumbersome, not readily available, and very expensive. Flame atomic absorption spectrophotometry is generally an excellent technique for the routine analysis of trace elements in the parts-per-million (ppm) range, but lacks the required sensitivity for measurement of manganese in tissues or fluids where the concentration of manganese is in the parts-per-billion (ppb) range.

Graphite furnace atomic absorption spectrophotometry, in contrast to flame atomic absorption spectrophotometry, has made available to investigators a method of measuring low concentrations of manganese (ppb range). This method is very sensitive and relatively inexpensive compared with X-ray fluorescence and neutron activation analysis. Despite the widespread use of this technique, there appears to be considerable inconsistency concerning sample preparation and choice of instrumental parameters. The latter problem is most likely a reflection of the variety of instruments commercially available. We have recently compared many of the more common wet and dry digestion procedures used for animal tissues, and have concluded that for most applications of flame atomic absorption spectrophotometry, wet digestion with nitric acid is the most suitable technique, providing elemental recoveries approaching 100% (3, 4).

We have now investigated the measurement of manganese by graphite furnace and flame atomic absorption spectrophotometry utilizing National Bureau of Standards (NBS) bovine liver as our reference standard. Results obtained from the graphite furnace methods are compared directly with results obtained by flame measurement as well as indirectly with the standard addition method. Our results are also compared with results published by NBS.

An important purpose of this study was to determine the ideal operational parameters for the "closed design" Instrument Laboratories 555B flameless atomizer. One operational parameter that has recently received a great deal of attention is the measurement of manganese concentration by either the peak height or by the peak area method. Sturgeon et al. (5, 6) have shown that peak area measurement may increase absolute sensitivity by a factor of two to eight, and extend the linear