



ACID VAPOR SAMPLE DECOMPOSITION IN OPEN VESSEL MICROWAVE

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Microwave decomposition has been increasingly used for a large number of samples. A method using an open vessel microwave was used for biological sample preparation with nitric acid vapors under atmospheric pressure.

A lab made Teflon® support suited with four cups, which received the samples, was inserted in the microwave vessel. These cups were used to weight 30 mg of certified reference materials such as spinach and apple leaves, which received 150 μ L of d.i. water to improve the microwave efficiency. This mixture was exposed to acid vapor stemmed from 15 mL of concentrated nitric acid placed in the bottom of the vessel for decomposition. The maximum microwave temperature, 115°C, was reached in 4 min. This condition was maintained for 20 min to allow complete decomposition of the sample. The decomposed sample, in the cup, was diluted to 1.0 mL of d.i. water, shaken and centrifuged. The cups were then placed directed into an autosampler for Co and Fe analysis in an atomic absorption graphite furnace spectrometer.

The proposed system allowed to minimize contamination, reagent and time consumption and the metal contents found were in accordance with the certified values for both metals.

Table 1. Analytical results for cobalt and iron in plant materials*.

| Reference material | Certified value (mg kg^{-1}) | | | |
|--|---|-------------------|----------------|----------------|
| | | Co | Fe | |
| Apple leaves (NIST 1515) | 0.09** | 0.08 \pm 0.03 | 80** | 83 \pm 4 |
| Spinach leaves (NIST 1570 ^a) | 0.39 \pm 0.05 | 0.31 \pm 0.03 | - | - |
| Corn kernel (NIST 8413) | 0.006** | 0.007 \pm 0.002 | 14.8 \pm 1.8 | 14.5 \pm 0.5 |

*Sample mass, 30 mg. The estimatives of uncertainties were based on three replications. ** Standard deviation not available.