

DETERMINATION OF SELENIUM IN ENRICHED AND NATURAL VEGETABLES USING SLURRY INTRODUCTION IN GF AAS

Rosa, C.R.*, Gomes Neto, J.A.*, Nóbrega, J.A.**. **Nogueira, A.R.A.*****

*Departamento de Química Analítica, Universidade Estadual Paulista, PO Box 355, 14801-970 Araraquara-SP, Brazil; **Departamento de Química, Universidade Federal de São Carlos, 13565-905, PO Box 676, São Carlos-SP, Brazil; ***Embrapa Pecuária Sudeste, São Carlos-SP, Brazil.

This work describes a method for the determination of Se in vegetables by GF AAS using slurry sampling. The electrothermal behavior of Se in 0.1% (v/v) HNO₃, vegetable digests containing 10% (v/v) HNO₃, or vegetable slurries containing 1% (w/v) plant material + 1% (v/v) HNO₃ in the absence and presence of modifiers Pd, Pd + Mg, Pd + Cd, Ni, Mg and Pd⁰ (pre-reduced) was investigated. Different sample pretreatment were also evaluated: the sample was dried by freezing in a high vacuum (lyophilization) or by heating in a stove at 65 C and thereafter digested (HNO₃ + H₂O₂) in open (block digester) and closed (microwave oven) systems. Additionally, dried samples were cryogenically powdered in order to prepare slurries: 1 g of powdered samples were dispersed in 100 ml of 1% (v/v) HNO₃ + 0.005% (v/v) Triton X-100. The flasks containing slurries were placed in a ultrasonic bath (30 min) for homogenization. A volume of *ca.* 1.5 ml of slurry was transferred to the autosampler cup. The slurry was manually homogenized by bubbling air inside it during the sampling by the capillary tube of the autosampler. Palladium nitrate (10 µg Pd) was used as chemical modifier and the following heating program of the Varian 880Z atomic absorption spectrometer was used: drying (85 C, 5s; 95 C, 40s; 120 C, 10); pyrolysis (1400 C, 13 s); atomization (2200 C, 3 s); tube clean (2600 C, 2 s). For 20 µL slurry, analytical curves in the 3.00 - 30.0 µg Se I¹ was obtained. The characteristic mass was 29 pg Se, the lifetime of the graphite tube was *ca.* 250 firings and the RSD (*n*=12) for a typical 15 µg Se I¹ was < 6%. Results for direct determination of Se in slurries of 12 vegetables were in agreement with those obtained for digests at 95% confidence level.