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CRYOGENIC GRINDING AND INFRARED HEATING AS ALTERNATIVES FOR SAMPLE PREPARATION. **Joaquim A. Nobrega**, Orlando Fatibello-Filho, Leticia M. Costa, and Maria Cristina C. Lombardi, Department of Chemistry, University Federal of Sao Carlos, P.O. Box 676, Sao Carlos, SP 13565-905, Brazil; Sandro T. Gouveia, University Federal of Ceara; Ana Rita A. Nogueira, Embrapa Pecuaria Sudeste, and Ramon M. Barnes, Department Chemistry, University of Massachusetts, Amherst, MA 01003-4310

Despite the advancement in sample preparation mainly propelled by the development of microwave furnace technology, sample digestion remains as the rate-determining step in the analytical sequence. For solid biological and botanical materials, comminution before the acid decomposition step is critically affected by the sample chemical composition. Samples with elevated contents of water, fibers, and fats are hardly reduced to small particles. Cryogenic grinding in liquid nitrogen is attractive, because practically all materials become brittle at this extremely low temperature.

In the developed work, a cryogenic grinder (Freezer Mill 6750, Spex Certiprep, USA) was employed for particle size reduction of breakfast cereals, raw vegetables, and corn. All these samples were completely comminuted in less than 3 min. Most particles generated were smaller than 37 μm. The samples pulverized were transferred to 10 mL closed borosilicate vessels and suspended in HNO_3 concentrated solution. The decomposition was performed applying infrared radiation. In the proposed procedure, 3 infrared lamps (15 V, 150 W each) were set in a triangular arrangement and the sample vessel was positioned in the focal point. The acid solution boiled in about 2 min when 10 V was applied to the lamps. After proper dilution, the solution or suspension obtained was completely suitable for introduction in ICP-AES (Varian, Vista Axial View) using a conventional concentric nebulizer. For comparison, samples were also prepared using conventional microwave-assisted, closed-vessel acid decomposition.

The results obtained for most metals, such as copper, manganese, iron, and zinc, measured using both sample preparation methods are in good agreement at the 95% confidence level (paired-t test). This work demonstrates that cryogenic grinding in liquid nitrogen and infrared radiation are fast procedures for particle size reduction and heating, respectively. Both the particle size obtained and the partial decomposition reached are totally compatible with sample requisites for ICP-AES.

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